Material change for a better environment

Final Report

Development of NIR Detectable Black Plastic Packaging

The aim of this project was to develop a solution to enable black plastic packaging that is currently destined for landfill or energy recovery, to be recycled. Currently the most common black pigments are based on carbon black and this packaging is not detectable by automatic Near infrared (NIR) sorting systems being used in plastics recycling. A wide range of potential solutions was investigated and viable options were identified in new machinery technologies and new colouration systems. Novel NIR detectable black colourants were developed and shown to work with APET, CPET, PP, HDPE, PS, and PVC in both laboratory and large-scale trials.

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Nextek Ltd

Front cover photography: NIR Detectable Black Trays

Executive Summary

This report describes the development of solutions that enable the mechanical recycling of black plastic packaging that is currently destined for landfill or energy recovery.

This research and development project was commissioned by WRAP to facilitate the development of technical solutions for the recovery and recycling of rigid black plastic packaging such as pots, tubs and trays from a mixed plastic waste stream and to deliver environmental benefits over existing waste management options.

Currently the most common black pigments are based on carbon black that is not detectable by automatic Near infrared (NIR) sorting systems being used in most Material Recovery Facilities (MRFs), Plastics Recovery Facilities (PRFs) and at reprocessors to separate plastics into different polymer streams for reprocessing into valuable materials. Carbon black is the name of a common black pigment, it appears black because it reflects almost no light in the visible part of the spectrum and also strongly absorbs in the ultra-violet (UV) and infrared (IR) spectral range. Carbon black is used as a colourant in food contact packaging for a number of reasons including that it provides a contrasting background and allows the colours in the food to stand out. It is low cost, has good dispersion and masking properties which allows off cuts of other colours to be mixed together and manufactured into black items.

NIR identification trials of commercially available black plastic packaging have confirmed that packaging coloured with carbon black cannot be detected by NIR systems and therefore typically ends up in the unsorted residue. It is estimated that there are around 30,000-60,000 tonnes per year of black plastic packaging in the UK household waste stream and although the On Pack Recycling Label (OPRL) for plastic pots, tubs and trays is “check local recycling”, there is a particular issue for carbon black packaging because even though it may be collected for recycling, there is currently a technical barrier to it actually being recycled back to virgin replacement resins within the UK.

Retailers are keen to address this current non-recyclable nature of black plastic and WRAP has worked in partnership with M&S, Sainsbury’s, packaging manufacturers and colourant suppliers on this project. The project has identified novel black colourants that are detectable by Near infrared (NIR) spectroscopy. The approach of using novel NIR detectable black colourants was shown to work with APET, CPET, PP, HDPE, PS, and PVC in both laboratory and large-scale trials at a PRF.

For the avoidance of doubt, the intention of the work was to find colouration systems and/or technologies capable of meeting the objectives of the project. No direct comparison of individual colouration systems or technologies can be drawn from the tables and information published throughout this report. The processes in this study were tested under different conditions, using different methodologies and are therefore not directly comparable.

This project explored four possible approaches to enable automated sorting of black plastic packaging. These were: alternative spectroscopic techniques, physical sorting methods, addition of detectable markers and the development of alternative colourants. Following a review and an evaluation of alternative spectroscopic techniques it was concluded that only the alternative colourant technique would allow sorting of black packaging with existing NIR based mixed plastics sorting facilities. Some alternative spectroscopic solutions such as Mid Infrared (MIR) were found to work on a technical level but are not yet viable at commercial sorting speeds. The project team therefore investigated solutions within the packaging and pigment industries that would work with current NIR sorting technologies.

The key principles utilised in identifying suitable black colourants were based on either of two characteristics; One: High levels of absorption of visible light and little absorption in the NIR spectrum, or two: Pigments which absorb in the visible spectrum but reflect in the NIR spectrum.

Pigments from both categories were identified from a range of suppliers and evaluated in this project. The range was reduced to a small number in order to evaluate the key principles, which should be equally applicable to other suppliers that can match the performance requirements as specified in this report.

The table below shows a summary of trials with alternative detectable black colourants and carbon black tested using NIR spectroscopic techniques. The recognition rate shows that these pigments allow the black articles to be readily sorted into polymer streams whereas carbon black coloured items could not be sorted.

Table 1: Summary of trial results for Sicopal, Lumogen, carbon black, Colour Tone and ColorMatrix colourants

	Detectable with NIR Spectroscopy?			**Average Recognition Rate**		
Polymer/Colourant	**PET**	**PP**	**PS**	**PET**	**PP**	**PS**
Sicopal K0095	✓	✓	✓	100%	100%	96.7%
Lumogen FK4210	✓	✓	✓	98.9%	100%	98.3%
Carbon Black UN MB	X	X	X	0%	0%	0%
Colour Tone IRR 95530	✓	✓	✓	97.6%	100%	Not tested
Colour Tone IRR 95550	✓	✓	✓	100%	Not tested	Not tested
ColorMatrix Dye Black-5	✓	✓	✓	100%	Not tested	Not tested

In large scale manufacturing trials at Sharpak, LINPAC Packaging Ltd and Faerch Plast, food contact trays were manufactured with PP, APET and CPET, using either the Colour Tone or the ColorMatrix colourants.

In general the sheet extrusion using the colourants selected was successful. All thermoforming operations ran well with no reported issues or changes needed from standard settings. Polypropylene and APET ran particularly well at all stages of the manufacturing process.

It is clear from the CPET trials that the colourants do need to be matched to the polymers and in commercial manufacturing operations this would indeed be the case. This would ensure colour depth is of a satisfactory level and that no colour bleed occurs. The grades of colourant eventually developed for CPET trays were subjected to severe tests designed to mimic cooking of oily food and they were found to pass, although early versions required further fine-tuning and formulation adjustment due to the types of tints being used.

For filling and sealing, laboratory sealing trials were completed on tray samples. In this project it was possible to seal all of the trays tested, although further large-scale trials would need to be completed to finalise the grade of lidding film for each pack type. NIR detectable colourants were found to have no significant difference to the heat required in achieving a seal.

The trays manufactured in the large-scale trials were taken to the Jayplas PRF in Derbyshire, which is a large sorting facility capable of separating the main packaging polymers by type and colour. In the sorting trial at Jayplas all the trays were correctly identified by the NIR detectors and were sorted into the correct polymer streams. However, the PET colour sorters seemed to be confused by the colour of the trays and sorted them into either the clear, clear and light blue or Jazz bunkers apparently at random. Subsequent analysis by the NIR sorting equipment manufacturer (TITECH) found that this would have been expected as the systems normally 'see' clear items as black when viewed against the black conveyor. They have confirmed that the sorting equipment software can be adjusted to segregate detectable black items from the clear items and this was tested and found to work well.

The alternative black colourants used in this project have been shown to allow effective sorting on existing NIR equipment with similar yields to clear packaging on a range of polymer types conventionally used for packaging materials.

Several other manufacturers of NIR sorting equipment were sent sample trays made from APET, CPET and PP, to determine whether the detectable pigments could be sorted on a range of commercial NIR systems. TITECH, S+S, RTT Unisort and Pellenc all reported that the black trays could be successfully and readily sorted into their respective polymer streams with high accuracy.

Recent evaluations have shown that the novel black colourants used in the large scale manufacturing trials are sufficiently stable for repeated reprocessing through conventional processes including extrusion at temperatures of 280 °C. It is important to note however, that the colourants developed do not have the masking strength of carbon black, which is related to the nano particle structure that characterises the carbon black colourants. This means that during recycling operations the colours that could be included in a black fraction may be limited to dark colours e.g. greens, blues and browns. Lighter colours, especially white, should be kept to minimal levels. Further studies are needed to identify which colours and the quantities of each that could be included without affecting the appearance of the recycled content product.

A cost assessment was carried out in order to provide a preliminary evaluation of financial viability of the alternative black pigments compared with the carbon black pigments. This would inform discussion with the industry and establish the case for further work. The indicative costs at various addition rates for the novel pigments has been calculated and compared to conventional carbon black masterbatch for a lightweight PP tray and a heavier CPET 'Ready meal' type tray. Additional costs may also be required for conversion of colourants in powder form to masterbatches to allow easy integration with existing manufacturing operations.

The detectable black colourants are more expensive than conventional carbon black pigments with an indicative price range similar to other specialised organic pigments of the order of £9 to £16 per kg.
The indicative increases in cost of using these alternative pigments in place of carbon black, is in the region of £1.80 to £3.50 per 1000 PET trays, at a 1% addition rate. For thinner PP trays this would be in the region of £0.75 to £1.50 per 1000 trays at a 1% addition rate. That's around 0.075 pence to 0.35 pence per tray at a 1% addition rate and implies an incremental cost per tonne of trays manufactured in the region of £70-£140.

However it should be noted that these indicative cost ranges are based on preliminary prices and it can be expected that if the supply chain wishes to implement alternative black pigments that commercial prices would be negotiated on the basis of large volumes, and therefore could be significantly lower.

Results from trials indicate that switching from a 1% carbon black addition rate to one of the new colourants may require an increase in addition rate to get the same depth of colour. A higher addition rate may also be required during reprocessing for improved masking capability. The cost comparisons for new product development need to consider the level of addition rate and this would need to be evaluated for each specific product as different addition rates may be needed depending on the product's wall thickness, etc. The cost differential will therefore be strongly influenced by the final addition rate and what is an acceptable black finish.

Although these alternative colourants do not match carbon black for tint strength or price at the moment, there are economic benefits arising from the recycling of up to an estimated 60,000 tpa of black plastic packaging through increased recovery yields and associated revenues and avoided landfill costs to the recycling industry. There are also opportunities across a number of other industries where it would be desirable to recover and recycle black items, such as electrical and electronic equipment (EEE), automotive and building/construction industries. These recycling operations would benefit from the increased recovery rates and higher throughputs that could be achieved with NIR sorting equipment.

Implementation of either detectable black colourants, or novel sorting technologies at PRFs that enable recovery of black plastic packaging is expected to have significant net environmental benefits over the current situation where black plastics are primarily ending up in landfill. Previous analysis by WRAP (LCA of Management Options for Mixed Waste Plastics) has found that approximately 0.6 tonnes of CO_2 can be saved for every tonne of mixed plastics mechanically recycled (WRAP, 2009).

In summary, this research has shown that a range of black colourants are now available to the plastics packaging industry and other sectors (including EEE and automotive) that would allow the automatic detection and recovery of these materials so that they can be used in new applications as substitutes for virgin plastics with subsequent improvements in recycling revenues as well as savings in carbon emissions and landfill volumes.
Further development work into these NIR visible colourants will in time allow economies of scale to be realised and improved masking properties to be developed.

Where possible, WRAP would recommend the use of alternative colours to black so that the pack can be detected and recycled. However, where a black colour is desired, WRAP would encourage retailers, brand owners and packaging convertors to investigate the use of NIR detectable black colouration systems further, with a view to them being widely adopted across the industry. The next steps need to involve retailers, brand owners and packaging convertors completing further trials on specific packs and carrying out detailed monitoring of specific products so that all issues relating to the economics, manufacture, filling, shelf stability and distribution could be fully validated.

In conjunction with this, further technical optimisation of the colouration systems could be conducted on the specific polymers and packaging systems to optimise the colours for specific packs and to improve the masking capabilities of the colourants to enable it to be recycled with a wider range of packs with other colours. This would have the potential to improve performance and may reduce costs through improvements in the scale of supply of specific masterbatches. As these commercially available pigments are taken up by the market, coordination and testing at major plastics MRF‟s and PRF‟s is recommended to ensure that the correct adjustments are made to the software used by the NIR sorting equipment at each facility, so that the black packaging is detected and directed into the correct recovery stream.

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Glossary

Acronyms used

APET	amorphous polyethylene terephthalate
CCD	charge-coupled device
CPET	crystallized PET, crystallized polyethylene terephthalate
GER	gross energy requirement
GWP	global warming potential
HDPE	high-density polyethylene
Jazz	mixed colour sorted plastic materials
LIBS / LIPS	laser induced breakdown spectroscopy / Laser induced plasma spectroscopy
MIR	mid-infrared
MRF	materials recovery facility
NIR	near infrared
RF	radio frequency
RFID	radio-frequency identification
PBT	poly (butylene terephthalate)
PE	polyethylene
PET	poly (ethylene terephthalate)
PP	polypropylene
PRF	plastics recovery facility
PS	polystyrene
PVC	poly (vinyl chloride)
PVDF	poly (vinylidene fluoride)
RDP	resource depletion potential
UV	ultra-violet
VIS	vision identification system
XRF	X-ray fluorescence

Spectroscopic method definitions:

LIBS (LIPS)	laser induced breakdown spectroscopy (laser induced plasma spectroscopy) measurement of the light emitted from plasma generated on irradiation with a laser
MIR	measurement of the absorption (or reflectance) of light in the mid-infrared region
NIR	measurement of the absorption (or reflectance) of light in the near infrared region
Photoacoustic	measurement of sound emitted from absorption of a light source modulated at audio frequency
Raman	measurement of the light emitted from Raman scattering induced by laser radiation
UV	measurement of the absorption (or reflectance) of light in the ultraviolet region
Visible	measurement of the absorption (or reflectance) of light in the visible region
XRF	measurement of the light emitted from the fluorescence induced by X-ray radiation

Other definitions:

Mixed Plastics	Non-bottle plastic packaging waste from households
Post-consumer packaging	Packaging collected after use by consumers
Triboelectric	Electrostatic charging of insulating materials

Acknowledgements

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1.0 Introduction

A significant portion of the plastic packaging used in the UK is coloured black. Black plastic is often chosen for food packaging as it enhances the appearance of the contents. Figure 1 shows a typical residue stream of rigid mixed plastics packaging after removal of PET and HDPE bottles.

Figure 1. Mixed plastic packaging including black plastics at a UK PRF facility

Black plastic packaging is most often made from polypropylene (PP) and crystallised PET (CPET), such as used in oven-ready meal trays. Trays made from amorphous black PET (APET) are also popular with UK retailers and can often contain post-consumer recycled content. Rigid black plastic packaging can also be made from polyethylene (PE), polystyrene (PS) and less frequently from poly vinyl chloride (PVC) polymers. The black plastic packaging currently being used cannot be detected and sorted in the normal automated systems used for sorting plastic packaging by polymer type, and manual sorting is not cost-effective. Consequently almost all post-consumer black packaging ends up in landfill.

Figure 2. Examples of black plastic packaging found in mixed plastic waste residue streams

1.1 Objectives of the project

The objective of the project was to enable black plastic packaging waste to be sorted by polymer type and recycled to produce high quality materials (as can be achieved with non-black plastic packaging) that can be used in place of virgin plastic to make new items. This would divert it from landfill and deliver savings in CO_2.

1.2 The black plastic tray waste stream

It is estimated that there are approximately 1 million tonnes of rigid mixed plastic packaging in the UK waste stream and the black plastic packaging could represent between 3-6% of this volume. Conservative industry estimates indicate that this could be around 26,000 - 30,000 tonnes per annum of black plastic packaging and estimates from other industry sources suggest that the figure could be as high as 60,000 tonnes per annum.

Therefore, in future, if recycling rates of black tray packaging matched the reported figures of current UK bottle recycling of 46%[1] there would be between around 11,000 – 27,600 tonnes per annum of this material diverted from landfill, so there is great potential in recovering this valuable resource.

2.0 Project Background

2.1 Sorting black post-consumer plastic packaging

The most common technology used for automated sorting of plastics uses near infrared (NIR) spectroscopy to detect the polymer type. These NIR detectors are capable of discriminating with good accuracy between containers and bottles made from different polymers such as PET, PP, PVC and PS (Blanco & Villarroya 2002).

Figure 3. Example of spectral fingerprints for different polymers in the NIR region (Source: TITECH GmbH)

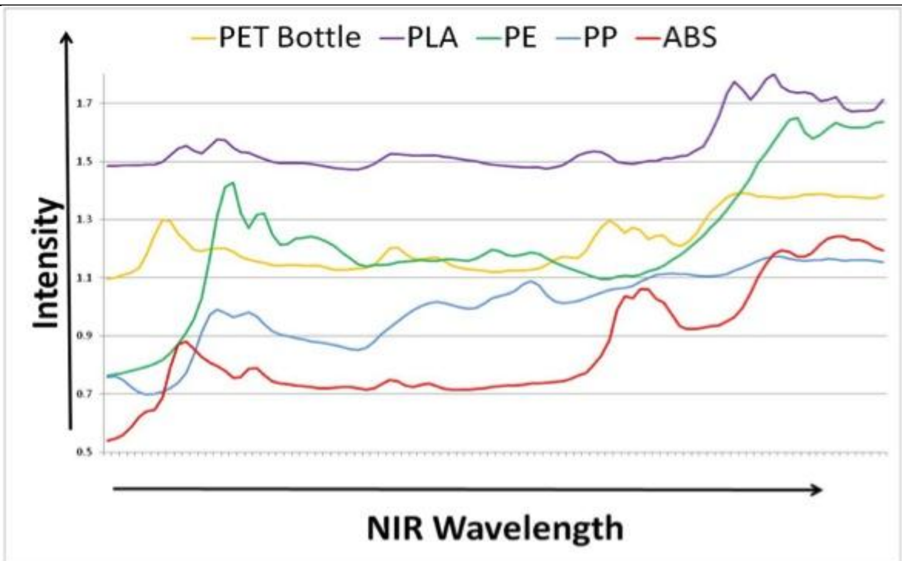


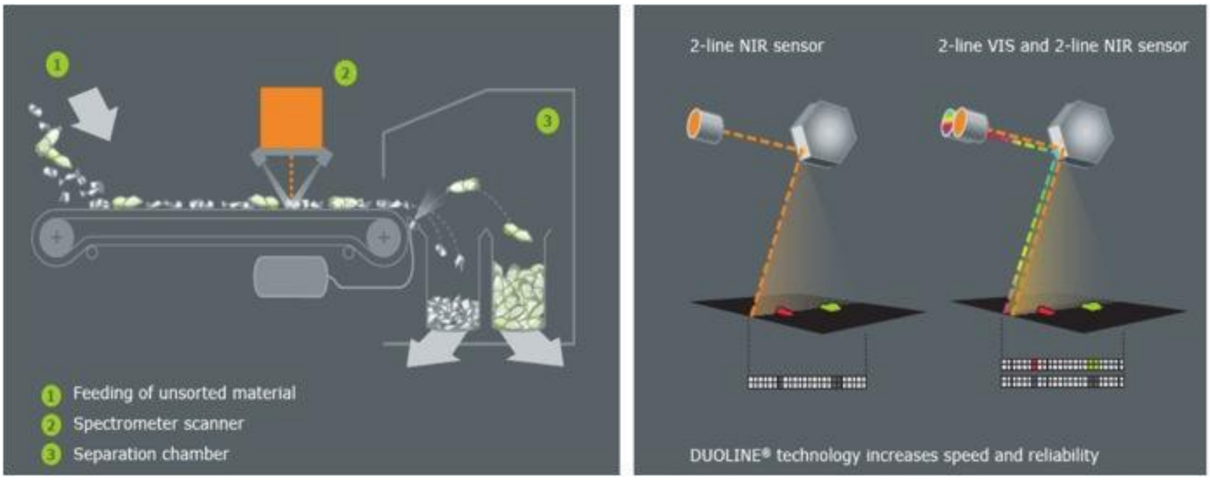
Figure 3 shows that each polymer has a specific and unique spectroscopic signature. This difference allows the NIR sensor to distinguish polymers from one another.

A typical sorting arrangement shown in Figure 4, involves the scanning of packages across a conveyor belt. As the articles pass under NIR detectors, the polymer specific spectrum of reflected NIR light received by the sensor is rapidly analysed by microprocessors and if a positive identification of the plastic type is made, the item can then be selectively ejected from the product stream with the use of air jets to a specified collection bunker.

Some sorting systems only perform polymer identification of a plastic packaging product, while others systems are designed to perform colour detection and polymer identification in one sorting step. This is shown in the right hand picture in figure 4.

[1] RECOUP, UK Household Plastics Packaging Collection Survey 2010, http://www.recoup.org/hppc2010/hppcsurvey2010.pdf

Figure 4. Example of packaging being sorted under NIR and VIS spectrum (Source: TITECH GmbH)

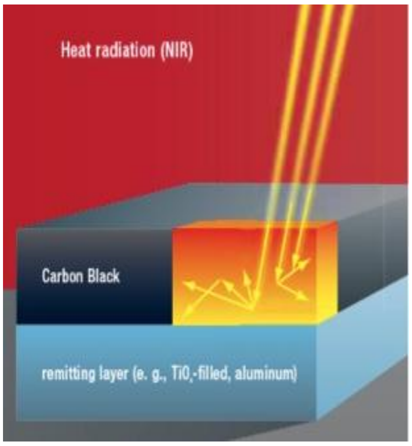


2.2 The recyclability of current black plastic packaging

Colourants in plastics can be either pigments or dyes. Pigments are finely divided, insoluble, inorganic or organic molecules, while dyes are organic light absorbing molecules that are soluble in the material (Christie 1994). The most common black colourant used in plastics is carbon black, as it is low in cost, has high tint strength, can be used in food contact packaging, and provides a contrasting background and allows the colours in the food to stand out. Carbon black pigments are produced by combustion of petrochemical feedstock, and they are available in a range of primary particle sizes, agglomerated structures and surface chemistries. All carbon blacks absorb strongly in the ultraviolet, visible and near infrared regions. Many other common black pigments, such as aniline, iron oxides and chromium oxides also have high absorption in the NIR region. Nigrosine blacks should have low absorbance in the NIR, but do not appear to be approved for food contact applications. Black antimony sulphide is a strong black pigment with fine particle size and IR reflectance properties (Murphy 2001). However, antimony compounds are toxic and restrictions apply to their use in plastics packaging.

Objects such as packaging products that have been coloured black do not emit or reflect in any part of the visible spectrum, i.e. they absorb all such frequencies of light. Pigments that absorb light rather than reflect it back to the eye "look black".

Figure 5. Strong NIR absorbing ability of Carbon Black (Source: BASF)



Black plastics that are coloured with carbon black colourants present a problem for NIR detectors as carbon black strongly absorbs infrared radiation as well as visible light, so the NIR light is not reflected into the detectors. This means that the items remain undetected and end up in the residual fraction from the sorting processes and are disposed of to landfill if this sorting method is used.

Separation of products that contain carbon black by colour is also not possible. This is because the strong absorbing properties of carbon black result in low signal reflectance and therefore the sensor cannot recognise the product’s colour.

2.3 OPRL labelling for black packaging

Currently the On Pack Recycling Label (OPRL)[2] used for plastic pots, tubs and trays is "check local recycling". However there is a particular issue for these packs that are coloured using carbon black because although they may be collected for recycling, there is currently technical barrier to them actually being recycled. There is currently a holding statement for black plastics:

'Black plastic packs: the current auto-sorting recycling equipment cannot identify black plastic. Where a dark pack is required or desired, a colour OTHER than black should be specified if possible. The position of black packaging is under review pending a technical project being run by WRAP to find a solution, and may change future OPRL Guidance.'

2.4 Delivery of the project

WRAP worked in partnership with M&S, Sainsbury's, packaging manufacturers and colourant suppliers on this project to address this current non-recyclable nature of black plastic. This report outlines the work undertaken, presents the results and puts forward the recommended next steps in order to make black plastic packaging more recyclable.

3.0 Possible Solutions for Recycling Black Plastic

This project explored four possible approaches to enable automated sorting of black plastic packaging. These were: alternative spectroscopic techniques, physical sorting methods, the addition of detectable markers, and the development of alternative colourants. The technologies reviewed are covered in detail in Appendix 1 and are summarised in the table below.

Table 2. Summary of potential approaches to sorting black plastics

Technology	Summary	Comments
Reflectance MIR and Laser Raman Fluorescence Spectroscopy	Has potential, but equipment limitations exist.	Systems available and being used for high speed sorting of black plastic flakes but not currently available for sorting of packaging articles such as pots tubs and trays
LIBS/LIPS	A very promising development for the future.	No commercial systems available.
Electrostatic separation	May provide adequate separation	Surface contamination can interfere with this system.
Tack separation	Will not supply sufficient separation for complex mixes.	
Alternative colourants	Black colourants with lower NIR absorption, such as BASF Lumogen and black IR-reflective colourants may work in reflectance NIR detector set-ups, but not in NIR transmission modes. Commercial IR-reflective black colourants such as BASF Sicopal K 0095 and trichromic mixtures of colourants or dyes provide black colouration without absorbing NIR strongly.	In all alternative colourants, food-contact approval and cost implications need to be examined. Low NIR absorption colourants may be detrimental to tray producers that thermoform from sheet due to longer heat-up times.
Addition of markers	Fluorescent markers could provide a cost effective method of identification.	The addition rates required to work effectively in black plastics need to be established.

Of these four approaches, alternative spectroscopic techniques and alternative colourants were selected for further investigation and developed further in this project. A subsequent review of spectroscopic technologies for detection of black plastics took place and is covered in Appendix 2. Following this it was concluded that only the alternative colourant technique would allow sorting of black trays with existing NIR based mixed plastics sorting

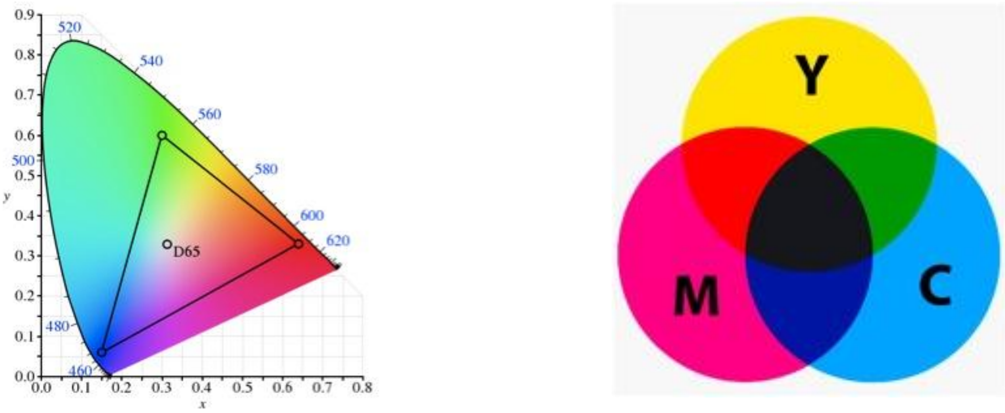
2 *http://www.onpackrecyclinglabel.org.uk/*

facilities. All of the other techniques would require investment in additional sorting equipment, ranging from an additional spectroscopic sorting stage through to reconfiguration of the plant to allow physical sorting of the out-throw stream. Given that alternative spectroscopic solutions such as MIR work on a technical level but are not yet viable at commercial sorting speeds and as most reprocessors and PRFs have NIR, a solution that is compatible with NIR is most likely to succeed in the short to medium term. As a consequence, the project team investigated solutions within the packaging and pigment industries that would work with current NIR sorting technologies.

4.0 Alternative NIR Detectable Black Colourants

A black pigment can result from a combination of several pigments that collectively absorb all colours. If appropriate proportions of three primary pigments are mixed, the result reflects so little light as to be called "black". Black can therefore be described as a lack of all colours of light, or a combination of multiple colours of pigment. Mixing yellow and cyan produces green colours; mixing yellow with magenta produces reds, and mixing magenta with cyan produces blues. In theory, mixing equal amounts of all three pigments should produce grey, resulting in black when all three are applied in sufficient density, but in practice they tend to produce muddy brown colours, or lighter shades of black.

Figure 6. Example of the RGB Colour Index and an example of how black colour can be achieved by mixing the key colours of magenta, yellow and cyan together (Source: The International Commission on Illumination)



It may be possible to select black colourants that absorb in the visible region, but reflect or transmit NIR radiation, allowing for the standard NIR detectors in automated sorting to register a useful spectrum from a black packaging article. However, the IR absorptive properties of standard carbon black colourants can be a benefit when thermoforming trays from plastic sheet because the higher infrared absorption increases the operating speed of the thermoforming equipment by enabling faster heat up of the sheet to moulding temperature.

4.1 NIR transparent colourants

Black colourants have been developed for use in coatings, paints and resins that act to reduce solar thermal gain in dark coloured building or automotive products by either being low in absorption, or reflective of infrared radiation. These colourants may prove to be suitable for black colouration in plastic packaging, yet enable detection by NIR spectroscopy. NIR transparent black colourants are used for laser welding plastic components. They allow infrared radiation to penetrate through one component to reach the interface with a second component that contains IR absorbers. The IR radiation causes local heating at the interface allowing the two components to be welded together. Examples of black colourants for these applications are the perylene colourants (BASF et al. 2005). These are organic perylene colourants that do not absorb strongly in the infrared region, but can be produced with high opacity and blackness.

A number of black colourants with high transparency in the NIR region were identified in a literature review:

- BASF Black NIR transparent colourants, such as Lumogen Black FK4280 and FK4281;
- Fuji Xerox developed colourants with high black colouration and NIR transparency for photocopy toner; and
- Treffert GmbH (www.treffert.org) produce specialised masterbatches for laser welding based on soluble dyes.

The BASF literature on Lumogen colourant shows significant transmission at wavelengths longer than 1000 nm. This is almost halfway in the near infrared spectrum (700nm-1400nm) as is defined by the International Commission on Illumination. BASF black colourant (Lumogen FK4281) is supplied to the building industry as it has low NIR absorption characteristic, which reduces solar heat gain. This colourant could therefore be used in packaging products, which could potentially be detected using NIR spectroscopy.

The Fuji Xerox patent application notes that they could also be used for laser welding of plastics (Hasegawa et al. 2009). They are based on a squarylium compound combined with phthalocyanine blue colourant. Treffert report that that have both laser absorbing and laser transparent types (Glaser 2006).

4.2 NIR reflective colourants

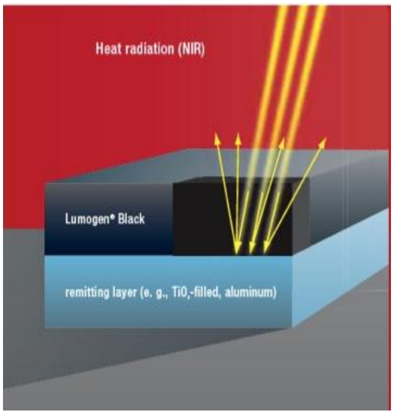
Paints and coatings with high infrared reflectivity or low absorption are now well known. They are useful for applications such as coatings for roofing tiles that enable dark colours to be used with less heat gain than normally achieved, and housings for lighting systems to reduce heat build-up.

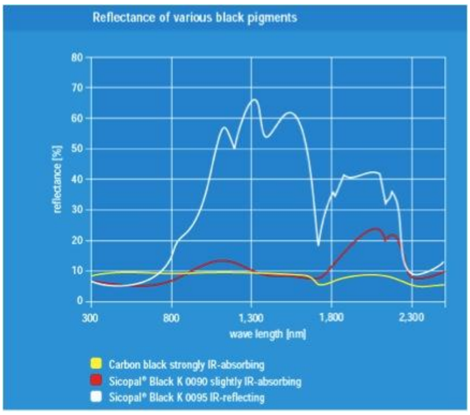
Several commercial examples exist of IR reflecting black colourants that can be used in plastics, such as Sicopal Black K0095 from BASF (also used in building products to help reflect sunlight) and Black 10P922 from Shepherd Colour Company. An IR reflective colourant at low concentration in the polymer matrix could allow for a useful infrared spectrum to be obtained in reflectance mode.

4.3 Review of alternative black colourants

Sicopal and Lumogen were compounded with PET, PP and PS at 0.1, 0.3, 0.5 and 1.0% addition rates. Plaque samples were made from different polymers using Lumogen FK4281 and Sicopal K0095 colourants at a range of colourant loadings and trialled at TITECH and Unisensor.

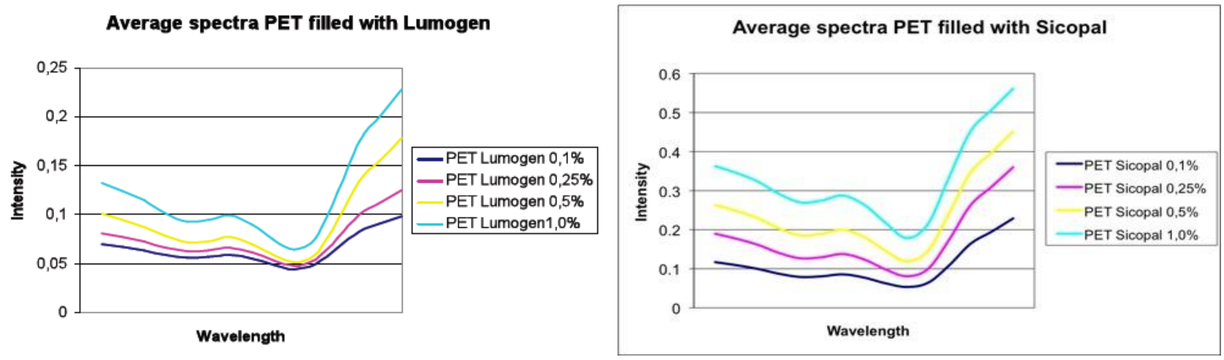
Figure 7. Picture showing reflective black colourants from BASF (Lumogen and Sicopal) (Source: BASF)





For comparison, a carbon black grade (Black UN MB) supplied by Begg & Co was also compounded with PET, PP and PS. NIR detection trials were performed on PET, PP and PS plaques coloured with varying levels of BASF Lumogen colourant. The following sections describe the spectra obtained and the detection rates for each polymer and colourant addition rate.

Figure 8. Average NIR spectra of PET filled with Lumogen and Sicopal



The average spectrum of PET with 0.1% colourant shows the lowest intensity. The reason for this is that the PET sample at this low addition rate is almost entirely translucent. Therefore the sensor detects PET and the black conveyor belt. The Lumogen and Sicopal samples with colourant concentration of 1.0 % show the highest intensity. In both cases the detection rate for PET was 100%.

4.4 Findings of preliminary trials of NIR detection of alternative colourants

The TITECH Autosort NIR sorting system was able to detect different plastics filled with Black Lumogen FK 4281 or Black Sicopal K0095 at different percentages. Samples that were coloured with a commercial carbon black grade called Black UN showed a random spectrum and/or very low intensity and could not be detected by means of NIR spectroscopy. Trials with another light source (43% increased intensity) still failed to detect the polymers coloured with Black UN, even with carbon black content down to 0.1%

Table 3. Summary of trial results for Lumogen, Sicopal and carbon black colourants

	Detectable with NIR Spectroscopy?			Average Recognition Rate		
Polymer	**PET**	**PP**	**PS**	**PET**	**PP**	**PS**
Sicopal K0095	✓	✓	✓	100%	100%	96.7%
Lumogen PET FK4210	✓	✓	✓	98.9%	100%	98.3%
Carbon Black UN MB	X	X	X	0%	0%	0%

The Unisensor, Powersort 200 system gave positive results for the Sicopal K0095 in all polymers, but the Lumogen FK4281 pigment in PP resin showed low spectra in the initial tests. Unisensor have reported that detection capability is now further advanced and detection is possible. Analysis by TITECH, using NIR of both Sicopal K0095 and Lumogen FK4281 gave readily identifiable black samples at 0.5 – 1.0% loading in all polymers tested.

5.0 Evaluation of NIR Detectable Colourants

Research of black pigments that reflect in the NIR spectrum but absorb in the visible spectrum identified potentially suitable pigments from BASF, Sun Chemicals, ColourTone and ColorMatrix. Colourants from these suppliers were compounded with typical packaging polymers at Begg & Co Ltd (shown in Table 4).

Table 4. Colourants compounded with packaging polymers into moulded plaques

Supplier	Colourant	Polymer
BASF	Lumogen AK	PP & PS
	Sicopal K0095	PP & PS
Sun Chemical pigments	Blue mixed with carbon black	PP
Colour Tone pigments	95491,95492,95493 and 95494	PP
ColorMatrix liquid colourants	Dye Black-5, Paul Black-1, Delphian Black-1 and Cimmerian Black-1	APET

The compounded materials were moulded into plaques at a loading of 0.5% and 1% and then tested for polymer detectability and also for colour detectability under NIR and VIS spectrum by TITECH. Table 5 shows the results that were obtained for PP, PS and APET. CPET material was not available for testing during the preliminary investigation but was tested using these colourants at a later stage and showed excellent processing performance and NIR detection.

Table 5. Summary of alternative black colourants tested by TITECH GmbH using NIR spectroscopic systems

Colourant Type & Addition Rate	Tray Material Type	Food Contact Certificate Available	Colour Detectable? (VIS)	Polymer Detectable? (NIR)
BASF Colourants				
BASF Sicopal K0095 (0.5%)	PP	TBC	x	✓
BASF Sicopal K0095 (0.5%)	PS	TBC	✓	✓
BASF Lumogen FK4281 (0.5%)	PP	YES	x	✓
BASF Lumogen FK4281 (0.5%)	PS	YES	x	✓
Sun Chemicals Colourant				
Sun Chemicals - 0.5% Total (0.25% Blue + 0.25% Black)	PP	YES	x	x
Sun Chemicals - 0.5% Total (0.375% Blue + 0.125% Black)	PP	YES	x	x
Sun Chemicals - 0.5% Total (0.125% Blue + 0.375% Black)	PP	YES	x	x
Colour Tone Colourants				
Colour Tone - Black 95491 (0.5%)	PE	YES	✓	✓
Colour Tone - Black 95492 (0.5%)	PE	YES	✓	✓
Colour Tone - Black 95491 (1%)	PP	YES	✓	✓
Colour Tone - Black 95492 (1%)	PP	YES	✓	✓
Colour Tone – Black 95493 (0.5%)	PP	YES	✓ / x	✓
Colour Tone – Black 95493 (1%)	PP	YES	✓ / x	✓
Colour Tone – Black 95494 (0.5%)	PP	YES	✓ / x	x
Colour Tone – Black 95494 (1%)	PP	YES	x	x
ColorMatrix Liquid Colourants				
ColorMatrix Dye Black-5 (0.5%)	APET	YES	✓ / x	✓
ColorMatrix Paul Black-1 (0.5%)	APET	YES	✓ / x	x
ColorMatrix Delphian Black-1 (0.5%)	APET	YES	✓ / x	✓ / x
ColorMatrix Cimmerian Black-1 (0.5%)	APET	YES	✓ / x	x

**Colour detection results marked with x means that the sensor sees the black tray as 'clear' e.g. as black conveyor belt or as 'unknown'.*
*** An NIR/VIS detection result marked as ✓ / x means that the sensor was able to detect the colour or polymer but system optimisation was required to achieve detection.*

The Sun Chemical formulations could not be detected, due to the carbon black content. It is very likely that this can be resolved with further investigation and use of another formulation.

The following list of colourants was found to enable the polymer to be readily detectable by NIR following tests with plaque samples and therefore provided the best opportunity for the recovery of black polymer packaging:

- PP/PE - Black 95491 (pigment) – Colour Tone Masterbatches Ltd;
- PP/PE - Black 95492 (pigment) – Colour Tone Masterbatches Ltd;
- PP/PE - Black 95493 (pigment) – Colour Tone Masterbatches Ltd;
- PP/PS – Lumogen AK (pigment) – BASF;
- PP/PS – Sicopal K0095 (pigment) – BASF; and
- APET - Dye Black-5 (liquid colourant) – ColorMatrix Group Inc.

Alternative colourants to carbon black that are NIR detectable are used for niche high-value applications due to their higher cost in comparison with carbon black. Plastic packaging for large volume items such as food trays requires pigments and colourants that are low cost. From a low cost point of view the following colourants appeared to be potentially economically viable (see section 13 for an economic assessment):

- PP/PE - Black 95493 (solid colourant) from Colour Tone Masterbatches Ltd;
- PP/PS – Sicopal K0095 (solid colourant) from BASF; and
- APET - Dye Black-5 (liquid colourant) from ColorMatrix Group Inc.

Food contact safety requirements have also been taken into consideration and during this research project all pigment suppliers were always asked to only supply food contact safe pigments. Certificates of compliance with EU and UK food contact regulations for selected colourants are provided in appendix 4 of this document:

- Black 95493 (pigment) – Colour Tone Masterbatches Ltd; and
- Dye Black-5 (liquid colourant) – ColorMatrix Group Inc.

5.1 Preliminary sheet extrusion & thermoforming trials

APET, PP and PS compounds were manufactured at Begg & Co and sent to Brunel University where they were extruded into sheet. The trials successfully produced PP and PS sheet but the processing equipment at Brunel University was found not suitable for the extrusion of APET sheet. Approximately 5 kg of PP and PS sheet was manufactured; the sheet samples appeared black and were measured to approximately 1.6mm thick. The laboratory-sized equipment did not permit the manufacture of thinner gauge sheet to allow comparison with commercially produced sheet samples, which are typically 0.5-1.5mm thick (depending on tray size and product application). The processing conditions and overall sheet extrusion performance using the alternative black colourants for the production of PP and PS sheet was found to be satisfactory and no problems were identified during the small scale trials.

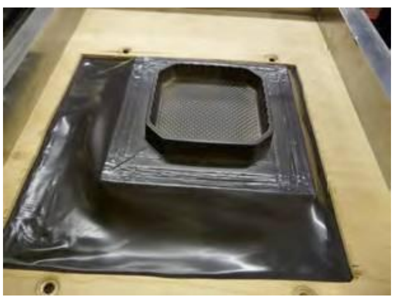
Figure 9. Example of sheet extrusion trials at Brunel University using alternative black colourants

The manufactured PP and PS sheet was then thermoformed by TMB Patterns (UK) to produce sample food trays. At TMB's thermoform tooling development facility, trials were performed under similar conditions to commercial systems and under standard heating times and temperatures to simulate similar cycle time performance. This approach provided very good small-scale trial data on the developed sheet thermoforming performance but it is not a substitute for actual large-scale performance trials as the trials were performed with a batch single cavity and not with multi-cavity continuous production.

Figure 10. Thermoforming cycle for production of sample trays

a) Heat soaked sheet

b) Upward movement of tool



c) Application of vacuum

During the thermoforming trials at TMB, 13 different compounds were run. Polymers tested included polypropylene (PP) and high impact polystyrene (HIPS). The trials showed that the cycle time for all materials was very similar to carbon black sheet and the heating and forming characteristics of the alternative black colourants were comparable. In summary, it was found that the colourants from BASF, Colour Tone and Sun Chemicals appear to absorb heat and thermoform in a similar way to standard carbon black sheet samples.

5.2 Further development and optimisation of alternative black colourants

Following the successful preliminary trials of colourants from BASF, ColourTone, and ColorMatrix, an expanded review of other suppliers of NIR reflective pigments was performed. More than 20 companies were spoken to about the development of NIR detectable black colourants and several colourant suppliers such as Holland Colours, Performance Masterbatches (PMB) and Silvergate Plastics had agreed to supply colourants that may reflect in the NIR spectrum and absorb in the visible spectrum.

Sheet extrusion trials using optimised colourant samples from ColorMatrix and ColourTone Masterbatches, and new samples from Holland Colours, Performance Masterbatches and Silvergate Plastics were performed at RAPRA using a small twin screw sheet extrusion line. Trials were performed using commercial grades of APET, CPET and PP resins supplied by Sharp Interpack and Faerch Plast. The colourants were added to the polymers at a range of addition rates suggested by the colourant suppliers. In all, over 30 formulations were extruded into sheet samples and these were subsequently thermoformed into trays at TMB Patterns. The sheet was extruded at nominal gauge thickness of approximately 0.7-0.8mm (700-800 micron) and the majority of colourants extruded and thermoformed without any significant problems.

Figure 11. Measurement of extruded sheet and examples of optimised colourants in thermoformed trays

a) Measurement of sheet thickness

b) Example of quality of trial trays produced

c) Finish of a CPET tray produced with 0.5% ColorMatrix Dye-Black-5

The trials found that there was a large variation in the blackness of the finish between the colourants tested. The following pictures show the trays manufactured.

Figure 12. Example of some of the PP trays manufactured with the colourants. A number of the trays were found to have acceptable black finish while others showed shades of dark brown, dark green and blue/purple.

This picture shows some of the black PP trays that were manufactured with the alternative black colourants. The picture was taken against a reflective white background to show the contrast in the colours obtained and the black finish achieved.

The trials clearly showed that some of the colourants did not absorb enough visible light and did not have enough tint strength in the visible spectrum as many trays appeared dark green, brown, various shades of blue. The tray shown in the middle was found to be closest to a black tray and was selected for large-scale trials. It was produced using the ColourTone IRR Black 95493 at 1% addition.

The APET and CPET tray samples produced also showed variation in colour and blackness of the finish (i.e. visible light absorption). Several colourants were tried with Colour Tone and ColorMatrix producing an acceptable black finish in aPET and cPET.

Figure 13. Example of some of the APET and CPET trays manufactured with the colourants

a) APET tray samples **b)** CPET tray samples

5.3 Summary of developed NIR detectable black colourants

In summary the NIR detectability of the PP and PET samples described in Section 5.2 was excellent with 100% polymer recognition of 26 of the 30 formulations tested. The colour detection of the NIR detectable trays was also found to be very good and the sorting system was able to classify the trays into a dark coloured stream. The 26 samples that gave favourable NIR and VIS spectrum detection results had varying pigment loadings of 0.3% up to 5%. The NIR and VIS spectrum tests showed that when a colourant was found to be detectable, the level of colourant loading in the polymer did not affect the NIR detectability of the polymer type of pack.

Figure 14. Example of PET tray produced with optimised NIR detectable black liquid colourant from ColorMatrix

From a visual perspective, the colour of some of the samples was not considered by participating retailers to be black enough for commercial applications.

Figure 15 shows an example of an NIR detectable black colourant, that was moulded into a plaque with varying thickness and the picture shows that the plaque appears sufficiently black in thicker sections but is clearly not black enough for thin wall applications due to the colourants low tint strength.

For this reason a number of the colourants were found not to be suitable for selection in large scale manufacturing trials. These colourants could however be optimised and may then be applicable for use in thin wall applications, such as plastic rigid trays.

Figure 15. Sample of PP plaque obtained during the development of an alternative black pigment showing low tint strength in thin sections. The thickness of the steps from left to right: 3mm, 2mm, 1.5mm, 1mm, 0.5mm

The trials also found that while many colourants appeared black in a masterbatch or compound granule format, once extruded and thermoformed into thin wall tray products, the tint strength of many of the colourants was not strong enough and the samples did not visually compare to black tray samples that were manufactured with carbon black.

From the extensive list of colourants tested a short list of most suitable colourants was selected for use in large scale manufacturing trials. The selection was based on the following criteria:

- Food grade certification;
- NIR and VIS detection performance;
- Colourant cost; and
- Black colour / shade (i.e. black finish).

The colourant systems were optimised through experimentation to improve tint strength and the colourants that were finally selected for large scale APET, CPET and PP were:

- ColourTone IRR Black 95530 for PP and CPET; and
- ColorMatrix Dye Black 5 for APET and CPET.

5.4 NIR identification and separation of APET and CPET packaging products

A key question arising from the development of detectable black APET and CPET trays was whether crystalline PET (CPET) and amorphous PET (APET) had unique spectra in the NIR region and if the two products could be separated using standard NIR sorting equipment. If the NIR detectable black colourants were to be adopted commercially then they would end up in the stream and there was potential of cross contamination of the coloured APET stream. CPET is highly crystallised polymer due to the presence of polyolefin nucleating agents and if mixed in with APET products during recycling, its presence could result in opacity during the production of APET sheet or PET bottles, which need to remain clear. APET however is not considered to be a contaminant in the CPET stream as CPET resin already contains APET along with the PE or PP nucleating agents that cause the rapid crystallisation of APET during the production stage.

In order to evaluate the feasibility of CPET and APET separation, commercial samples of white CPET were obtained from Faerch Plast (commercial black CPET samples cannot be detected under NIR) and were sent to TITECH together with several CPET trays produced with the newly developed NIR detectable black colourants.

TITECH performed several identification tests and found that there were sufficient spectral differences between APET and CPET to allow efficient sorting. A dynamic sorting trial was performed to separate the white CPET trays and CPET with NIR detectable black colourants from clear PET bottles.

Figure 16. Results from an APET / CPET sorting trial - showing good separation of CPET from clear APET

a) Clear PET bottles

b) Commercially available white CPET trays and black CPET trays made with the novel NIR detectable colourants

The results from the sorting trials indicated that it is technically possible to separate CPET from APET based on spectral differences. TITECH reported that slight changes to the sorting software programmes and 'sensor training' would be all that is needed to make this change in commercial TITECH systems currently in some PRFs that may want to sort CPET. A key question for future research and development is whether it is possible for NIR detectable black APET and CPET trays to be mixed and re-formulated to produce a 100% rPET CPET resin. Further validation would be required to ascertain whether other NIR sorting equipment can easily be adjusted to enable them to distinguish and sort CPET from APET.

6.0 Industrial Scale Manufacturing Trials

Following the development of two colouration systems that were NIR detectable and had been optimised in terms of their shade of jet blackness, the next step in development was to complete large scale manufacturing trials. The purpose of these trials was to assess the processability of the pigments on industrial scale machinery and to assess whether there was any difference in heat settings or dwell times needed to manufacture products using the NIR visible colourants compared to products manufactured using carbon black. The large-scale trials were used to observe the heat absorption characteristics of the newly developed alternative black colourants and to determine thermoforming performance under high speed commercial production rates. Carbon black strongly absorbs the infrared energy used in thermoforming and the absence of carbon black might cause longer sheet reheat times. Data from these trials would play an important role in establishing whether sheet containing the alternative black colourants (which do not strongly absorb infrared energy) performed adequately under thermoforming conditions and if they absorbed heat at similar rates as sheet containing carbon black.

The following tray manufacturing companies who have extrusion and thermoforming capability agreed to be involved and run a manufacturing trial using at least one of the selected colourants and one polymer type to cover PP, APET and CPET since together these polymer groups make up to 80% of the black plastic packaging market and almost all are used in food contact applications.

Table 6: Summary of industrial scale manufacturing trials

Manufacturer	Material	Colourant	Recommended addition rate (%)	Addition rate used (%)
Sharp Interpack	PP	Colour Tone IRR 95530	2%	2%
LINPAC Packaging Limited	APET	ColorMatrix Dye Black-5	0.5%	0.8%
Faerch Plast	CPET	Colour Tone IRR 95530	2%	4%
Faerch Plast	CPET	ColorMatrix Dye Black-5	2%	4%

Faerch Plast added 4% masterbatch because they believed that amount would be needed to match the blackness of carbon black.

6.1 Manufacture of polypropylene trays

Sharp Interpack extruded PP sheet using virgin polymer and added the Colour Tone IRR 95530 black masterbatch at 2% addition rate. This was then converted by thermoforming into approximately 10,000 trays using 50 kg of PP resin. The tray manufactured by Sharp Interpack was a Marks and Spencer's mushroom punnet.

Figure 17. Polypropylene trays moulded by Sharp Interpack using Colour Tone IRR black 95530 masterbatch

During the trials the machine settings were monitored and no changes were made from the standard settings used for processing black PP coloured with carbon black and no processing issues were encountered.

The finished trays pictured above had a commercially acceptable black colour and were accepted by two major retailers based on the shade of black.

6.2 Manufacture of amorphous PET trays

LINPAC Packaging manufactured 600 micron thick APET sheet using Dye Black 5 liquid colourant supplied by ColorMatrix. Since LINPAC Packaging does not normally use liquid colourants, a dosing unit was supplied by ColorMatrix to help facilitate the trial.

Once the dosing unit was installed the line was run until clear sheet was being extruded to purge the system of any residual carbon black colourant. The liquid dosing unit was then switched on and set to 0.5%. This was the target figure based on lab scale trial samples that had been approved by the retailers. However, once the sheet was thermoformed the colour was much paler than the sample to be matched, so the addition rate was increased to 0.8%.

Figure 18. Pictures from APET sheet extrusion and thermoforming trials at LINPAC Packaging

a) Liquid colourant dosing unit for the Dye Black-5 colourant supplied by ColorMatrix

b) Picture showing the alternative black colour coming through the extruder at LINPAC Packaging

c) Black APET trays being thermoformed at LINPAC Packaging

Figure 19. Example of visual appearance of APET trays produced when held against bright light and without back lighting

a) APET samples showing difference between 0.5% (top) and 0.8% (bottom) colourant addition rate when held against bright light

b) Manufactured APET sample photographed on a shelf showing no translucence

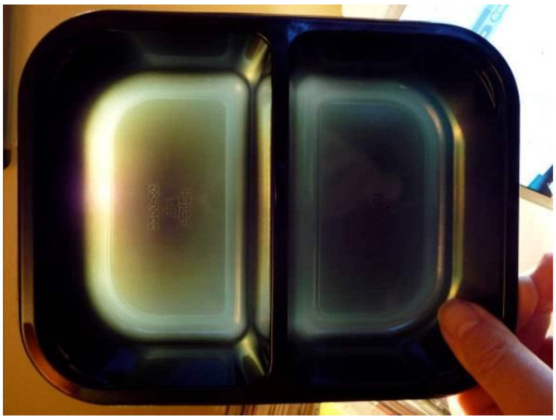
During the trials the process settings on both the extrusion and the thermoforming systems remained unchanged from the conditions used to process carbon black material. No processing issues were identified during this trial. The tray manufactured was a generic UK food tray manufactured in 400μm APET with 35μm PE overlaminate and approximately 3,000 units were produced for testing in large scale recycling trials.

6.3 Manufacture of Crystalline PET trays

Faerch Plast in Denmark is a major supplier of CPET packaging to the ready meals market in the UK and agreed to carry out two CPET manufacturing trials. The first trial used the masterbatch supplied by Colour Tone IRR 95530 and produced 3,700 thermoformed bowl units (Figure 20a). The second trial used a masterbatch version of the ColorMatrix liquid colourant Dye Black-5 and produced 3,400 tray units (Figure 20b).

Figure 20. Example of visual appearance of CPET trays produced without back lighting and when held against bright light

a) CPET bowl (C0160-1D) manufactured using Colour Tone IRR 95530 by Faerch Plast



b) CPET tray (C2200-2G) manufactured using ColorMatrix.
Dye Black 5 by Faerch Plast (with back lighting)

The IRR black 95530 from Colour Tone had originally been formulated for PP and was made using a PE base for the masterbatch. At higher processing temperatures (300°C) for CPET some thermal degradation of the masterbatch system was observed. This degradation occurred because the processing temperature of CPET is higher than that used for PP and in this instance was higher than the expected temperature of 260 °C up to which the colourant was thermally stable. The trial observations and comments from production staff on this trial were that the Colour Tone IRR 95530 gave a visually bad sheet with a lot of specks and that there were difficulties in the extrusion process including a red deposit on the chill rolls. The presence of specks was related to the degradation of the PE based masterbatch material. If the Colour Tone masterbatch had been developed for PET and designed to be thermally stable up to temperatures of 300 °C then this degradation would not have occurred. It is possible the red deposits were carry over from another trial completed at the site before Faerch Plast ran this trial and may in-fact be unrelated to the use of IRR 95530. Thermoforming performance of the round bowl (0160-1D) was found to be satisfactory and no processing issues were encountered.

The second CPET trial involved the manufacture of a two compartment tray (2200-2G) and was performed using a PET based solid masterbatch prepared from the ColorMatrix Dye Black-5 liquid colourant. The observations and comments from this trial were that extrusion and thermoforming processes were satisfactory, but there was insufficient colour strength.

The approved masterbatch samples from ColourTone and ColorMatrix for these combinations were at 2% addition rate and this was the planned addition rate for the trials. However the operators at Faerch Plast were requested to use their experience on the addition rate level and if it was felt that the colour was too weak, they were asked to increase the level. As this was the case in both trials Faerch Plast ran both colourants at 4% addition rate in the CPET trays.

Lower addition rates could be achieved in future product developments through further optimisation of the colourants for specific packs and customer approval of newly developed packs, which may have a variety of black shades.

6.4 Colour migration

Colour migration tests designed to simulate ready meal cooking conditions were carried out by Faerch Plast on the two CPET tray types. Ideally there should be no migration. The results showed that some colour migrated from the polymer matrix into vegetable oil at 180 °C after 30 minutes. The image below (Figure 21) shows the oil samples from the migration trials, with the control sample in the middle, the Colour Tone IRR 95530 sample on the left and the ColorMatrix Dye Black-5 sample on the right.

Figure 21. Colour migration into oil from CPET trays after 30 minutes at 180 °C showing the Colour Tone IRR 95530 sample (left) the control sample (middle) and the ColorMatrix Dye Black-5 sample (right)

The soluble Dye Black 5 supplied by ColorMatrix (right hand sample) showed a high level of colour migration in this test and its current formulation is unlikely to be suitable for use in a crystalline structure like CPET. The less soluble colourant IRR 95530 supplied by Colour Tone (left hand sample) showed mild colour migration, likely due to it not being optimised for CPET resin.

Tests on the ColorMatrix colourant in the APET and PP samples at ambient temperature (20 – 25°C) for five days have shown no change in the colour of the oil, see Figure 22 below.

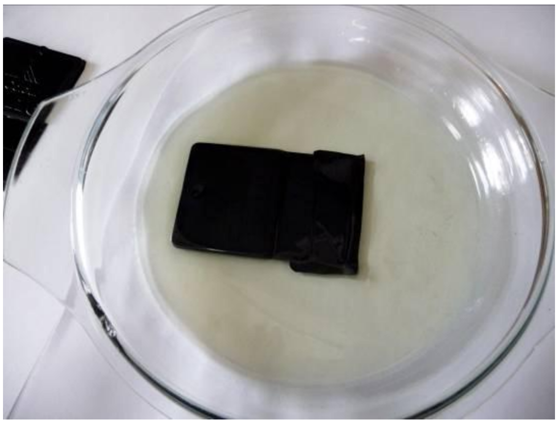
Figure 22. Oil samples showing no colour migration from APET sample with ColorMatrix Dye Black-5 (left), the control sample (bottom) and the PP sample with Colour Tone IRR 95530 (right) after storage for five days under ambient conditions (20 – 25 °C)

In further developments, Colour Tone identified the component of the colour system that was found to be bleeding from the CPET tray during the hot oil tests and reported it to be an organic dye component, which was independent of the NIR detectable colour. This component was replaced with another and the new colourant called IRR 95559 was tested for 30 minutes in oil in an oven at 180 °C and did not bleed. Figure 23a shows PET plaques at 1% addition rate and 3% addition rate. Figure 23b shows a PET plaque with 3% addition of the IRR

95559 after 30 minutes in oil in an oven at 180°C. No colour bleed from the PET plaque into the hot oil was evident after 30 minutes.

Figure 23. Optimised Colour Tone colourant (IRR 95559) for PET and CPET showing PET plaques at 1% and 3% addition rate (left) and a PET plaque with 3% IRR 95559 addition showing no colour bleed from the PET plaque into oil after 30 minutes at 180°C (right)

a) Optimised Plaques for PET (Colour Tone)



b) Optimised plaque after 30 minutes in oil at 180°C

ColorMatrix has reported that it is planning to develop a solution for CPET resin for use in ready meal packaging.

6.5 Summary of manufacturing trials using NIR detectable black colourants

In general the sheet extrusion using the colourants selected was successful. All thermoforming operations ran well with no reported issues or processing changes needed from standard settings. The reported colourant dispersion issues in CPET can be explained by the use of a less suitable polymer carrier for the masterbatch. Later optimisation showed that the colourant was successfully used in PET resin and once it was re-formulated it was found to disperse well within the PET resin and all components were thermally stable at the higher processing temperatures.

Both the Colour Tone IRR black 95530 colourant used in the PP resin and the ColorMatrix Dye Black-5 liquid colourant used in APET performed particularly well at all stages of the tray manufacturing process.

It is clear from the CPET trials that the colourants do need to be matched to the polymers and in full-scale manufacturing this would indeed be the case. This would ensure colour depth is of a satisfactory level and that no colour bleed occurs.

The issue of colour bleeding from the CPET trays when exposed to hot oil conditions was initially of concern. Further optimisation work on the Colour Tone masterbatch components has resolved this issue by successfully replacing the component that bled out during the hot oil colour migration tests with a component that did not migrate out of the polymer matrix. The current ColorMatrix Dye Black-5 formulation appears to be unsuitable for use in a polymer with a highly crystalline structure like CPET, however ColorMatrix believe it can develop a suitable alternative black colourant for use in CPET applications that will be stable, will not migrate into oil under hot conditions and will be NIR detectable.

Figure 24. Suitability of colourants in PP, APET, CPET

POLYMER	COLORMATRIX DYE BLACK 5	COLOUR TONE IRR 95530	COLOUR TONE IRR 95559
PP	-	✓	-
APET	✓	-	✓
CPET	x	x	✓

6.6 Conclusions

Two colourant systems selected for development and large scale manufacturing trials were a liquid colourant from ColorMatrix - Dye Black 5 that had been previously tested in APET and a solid masterbatch from Colour Tone - IRR Black 95530, previously tested in PP.

Sharp Interpack in Yate successfully ran a tray manufacturing trial using a masterbatch colourant from Colour Tone at 2% addition rate into PP. Sharp produced 10,000 PP mushroom punnets and found that there were no processing issues during the trial and that none of the machine settings needed to be changed from the presets used when running carbon black as a colourant in PP sheet.

LINPAC Packaging at Featherstone installed a liquid dosing system and trialled a liquid colourant from Colour Matrix. LINPAC manufactured over 3,000 APET trays starting at an addition rate of 0.5% that was increased to 0.8% to give a satisfactory depth of colour. No process settings were changed from those used when processing carbon black trays and no concerns were identified during this trial.

Faerch Plast in Denmark ran two manufacturing trials in CPET. They used a solid version of the Colour Matrix Dye Black 5 manufactured specially for the project by ColorMatrix and solid PE based masterbatch from Colour Tone. Since neither of these colourants had been tested in CPET the manufacturer was asked to start addition at 2% but if needed they could increase the dosing level to a satisfactory level. Faerch Plast reported problems with the PE based Colour Tone masterbatch producing specks in the sheet during extrusion and depositing on the rollers. ColourTone later reformulated the masterbatch for use in PET and the dispersion issue was resolved. The extrusion of the Colour Matrix material did not cause any issues and both materials thermoformed well.

From the manufacturing trials it is concluded that ColorMatrix Dye Black-5 is suitable for use in APET and other ambient applications but would not be suitable for use in oven ready applications where the food is cooked in the packaging. The current ColorMatrix Dye Black-5 formulation appears to be unsuitable for use in a polymer with a highly crystalline structure like CPET, and ColorMatrix is planning to develop a suitable alternative black colourant for use in CPET applications that will be stable, will not migrate into oil under hot conditions and will be NIR detectable.

Colour Tone's IRR95530 was suitable for PP tray applications but was not suitable for use in CPET ready meal products. Colour Tone developed an improved colourant (IRR95559), by removing one of the components found to be bleeding and replacing it with a stable component that resulted in the optimised colourant being suitable for use in PP, APET and CPET applications including oven ready applications. Whilst the bleeding/colour migration property of the re-formulated pigment was tested in plaque form, a sample tray was not produced and would need to be completed as a next step to ensure that this pigment looks acceptable when thermoformed into a tray. Further optimisation may be required to ensure this is aesthetically acceptable in tray form.

It is clear from these manufacturing trials that newly developed colourants need to be specifically formulated and matched to the polymers and tests need to ensure a satisfactory level of colour depth and that no colour bleeding occurs in specific packaging applications.

7.0 Filling / Sealing trials

In order to test whether the use of the NIR detectable colourants that had been developed for APET, CPET and PP tray packaging had any effect on the heat-sealing performance of trays, small scale sealing trials were performed using standard manufacturing operations.

It was assumed that the mechanical properties of the tray samples had not been altered by the colourants so actual filling and storage trials were not conducted. The use of alternative colourants does not change mechanical properties such as tray top load strength as the pack mechanical properties are obtained from the polymer and tray design and not the colourant. Yellow rice was added to the tray samples prior to the heat sealing trials to enhance the images.

The aim of the trial was to assess the amount of heat and dwell time needed in the sealing machine to impart a satisfactory seal and compare these parameters against a standard pack of the same type coloured with carbon black, and to assess the visual appearance of the filled and sealed tray.

Samples of the APET and CPET trays were sent to Pennine Foods for filling and sealing trials. Using a small continuous production line with the APET trays it was not possible to get the lidding film to seal correctly. This could be due to a number of factors but was attributed to the fact that Pennine foods do not run this particular tray size and shape and so didn't have the correct tooling set to seal the tray correctly under the sealing head.

Laboratory sealing trials were then completed using a hand held sealer. The APET and CPET tray samples were heat sealed with a lidding film, usually used for PP trays but it was found that the APET and CPET trays could be sealed with this lidding film. In the manual trials it was possible to seal all of the trays, although it is recommended that further trials would need to be completed to identify the optimum type of lidding film for each pack type. From the manual sealing it appears that the NIR detectable colourants made no difference to the heat or the dwell time in the sealing machine required in achieve a functional seal.

Figure 25. The images below are from the filling and sealing trials completed at Pennine Foods. The sample shown bottom right is the standard tray coloured with carbon black and used as a control to compare sealing

a) Sealed APET tray

b) Sealed CPET bowl

c) Sealed two compartment CPET tray

d) Top view of sealed APET tray

e) Top view of sealed CPET bowl

f) Sealed carbon black control sample two compartment CPET tray

7.1 Conclusion

Trials completed on the APET and CPET samples show that sealing can be achieved satisfactorily and that no additional heat or extended dwell time is needed for packaging coloured with detectable black colourants.

Sharp Interpack also sealed sample punnets and took photographs. The samples were both filled with 250g organic closed cup mushrooms.

The samples were:

1. Standard Polypropylene blend plus 2% carbon black masterbatch IRR.
2. Polypropylene blend plus 2% ColourTone IRR Black 95530.

The film was a standard easy-peel PP film, and there were no issues sealing to either punnet. The sealing machine was a lab model, and so it was not possible to seal at production speeds. The main point was that there were no differences between the 2 punnets. As can be seen below, they both looked black.

Figure 26. The images below are from the filling and sealing trials completed at Sharp Interpak (the photograph shows 2 punnets, the IRR is on the left side)

8.0 Industrial Scale Identification and Sorting Trials

In order to demonstrate on a commercial scale recycling operation whether the use of the developed colourants would indeed enable the packaging to be sorted by NIR and VIS sorters a large scale trial was carried out at the Jayplas plastics sorting plant in South Normanton. The trials completed at Jayplas were performed to assess industrial NIR detectors ability to sense and sort the black packaging samples manufactured with NIR detectable colourants.

Industrial scale sorting plants are generally configured to focus on the material types that give the biggest yield and are of commercial value to the operator. This means that plants can vary considerably by design. The Jayplas facility is capable of separating the major polymers within a mixed plastic stream by polymer type and colour. With a capacity of 78,000 tonnes, the facility sorts HDPE, PET, PP, PS and PVC mixed plastic bottle stream containing approximately 20% other rigid plastics. The materials are collected from local authorities, waste management companies and large national supermarkets.

At Jayplas, baled plastics are put through a rigorous sorting process, removing any contaminants such as paper and plastic bags. The remaining plastic material is divided by polymer type and colour using NIR and VIS optical sorting technology from TITECH that it is able to divide HDPE and PET by colour into natural, blue tint, blue, green and mixed. The sorting systems analyse all plastics with a fast-moving scanning sensor fitted over the conveyor belt, which identifies material types and colours as well as the object position on the conveyor belt in a split second. Once the sensor has defined the plastic pack polymer type or colour, the plastic pack is blown onto the appropriate conveyor using high power air jets, while the non-plastic waste is delivered to a third belt for further sorting. The end product, having been sorted by colour and type, is re-baled for further processing off site.

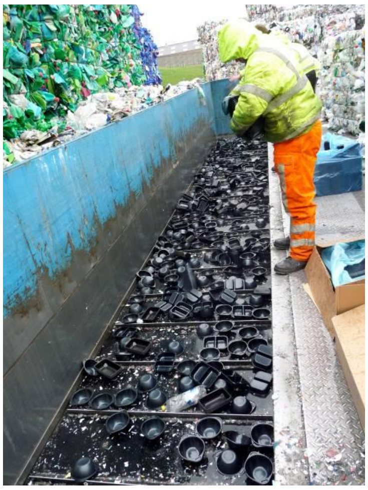
Table 7. Sorting steps from a typical PRF

Sorting Step	PET Sorting Detector type	Sorted Products (PET)	Sorted Products (Non PET)	Non PET Sorting Detector type
1st sort	NIR	PET	HDPE & other plastics	NIR
2nd sort	NIR & Colour	Clear PET	HDPE	NIR
3rd sort	Colour	Clear PET	PP	NR
4th sort	Colour	Green PET	Others as required	NIR & Colour
5th sort	NIR	Jazz PET		
Negative sort	-			

Approximately 163 kg of the APET (51 kg), CPET (72 kg) and PP (40 kg) trays produced in the manufacturing trials were scattered across the infeed belt and then covered with approximately 1,700 kg of mixed plastic bottles.

This gave approximately 9% concentration of black trays within the mixed plastics stream after the ballistic separators to simulate the amount of black plastic currently found in a mixed stream. The facility currently receives approximately 7-10% mixed black plastic packaging. This material cannot be recycled and is either landfilled or sold as part of a residual mixed plastics fraction.

Figure 27. Preparation of materials for sorting trials at the Jayplas PRF

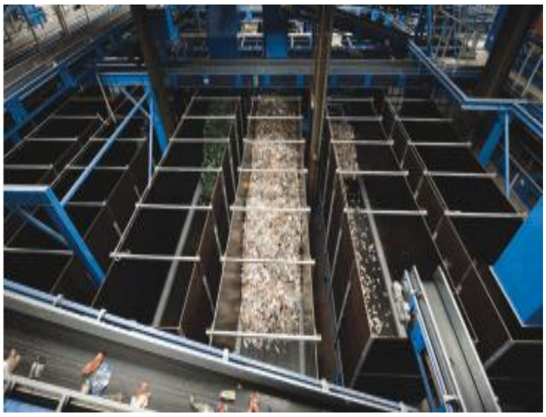


a) Black plastic trays being distributed on the sorting plant in-feed conveyor

b) Mixing the trays with mixed bottles prior to sorting

c) TITECH NIR sorters

d) NIR detectable black trays on the first sorting belt



e) HDPE packaging collection bunker

8.1 Polypropylene sorting trials

At the Jayplas facility PP products are sorted from other polymers into a PP bunker. Currently Jayplas only sort PP by polymer type into a mixed colour PP bunker. The mixed PP bales include natural and all other colours (except black which can't currently be identified).

Figure 28. PP trays in the PP bunker after sorting

It is estimated that over 99% of the black PP trays with the optimised Colour Tone pigment that were put in at the start of the sorting process were correctly identified as PP. The black PP trays were sorted into the mixed colour PP bunker. Jayplas do not sort the PP into separate colour streams as there is not enough natural PP to warrant natural PP and coloured PP separation.

Section 9 of this report shows that it is possible to separate the alternative black PP trays away from natural and into a coloured PP stream if recovered natural PP packaging volumes of were to increase.

8.2 APET and CPET tray sorting trials

The first NIR sort step at the facility involved positive identification and separation of PET packaging from all other materials. The first colour sort for PET was performed to separate clear PET. It was found that approximately 10% of the black PET trays were in this bunker and of those most were the round CPET trays, manufactured by Faerch Plast using the IRR 95530 black colourant from Colour Tone.

The next colour sort involved separation of clear and light blue PET. In this bunker it was estimated that 30% of the black PET trays were found here, again mainly the round CPET trays.

The next sort was again a colour sort and this separation stage involved separation of green PET. It was estimated that less than 1% of the trial black trays were found in this bunker.

The final PET line sort was a positive NIR sort designed to recover Jazz (mixed coloured) PET, ie all the PET missed by previous sorts. It was here that it was expected to recover all the NIR detectable black trays. In this trial it was found that around 50% of the trial trays were in this bunker and those were mainly the rectangular APET tray and the two compartment CPET trays.

Figure 29. PET sorting stages at the Jayplas PRF

a) First sort - Clear PET bunker

b) Second sort - Clear and light blue PET bunker

c) Fourth sort - Jazz PET bunker

d) Residue "rests" bunker showing no loss of NIR detectable black PET

The final bunker inspected, as part of this trial was the residual 'rests' bunker, which contained all the items not identified by the previous sorts. The residual materials included difficult to identify and sort items such as fines and bottle 'rollers' filled with air, which don't get ejected because they roll away from the air jets.

Some black trays were found in this bunker but these were identified as standard carbon black trays and not the NIR detectable black trays tested in this trial. It was calculated that less than 1% of the trial trays were in this bunker, meaning that 99% of the NIR detectable APET and CPET trays were successfully sorted by polymer into the PET stream.

8.3 Sorting trial summary

In the large scale sorting trials virtually all the PP and PET trays (>99%) were correctly identified by the NIR detectors as PP and PET and were successfully sorted into the correct stream on the basis of polymer type. The PP trays were only sorted by polymer type as Jayplas do not sort PP by colour due to lack of natural PP in the currently collected packaging waste stream. However, they do sort clear and coloured PET into separate streams, and the trials showed that the PET colour sorters were inadvertently directing black trays into either the clear, clear and light blue or jazz bunkers. The reason why the black PP and PET trays did not go into the respective coloured (jazz) PET bunkers was because the software on the sorting system at Jayplas was not set up to be able to identify black colour and the sensors 'saw' the black trays as clear/natural when scanned against a black conveyor belt background. This issue was later overcome by TITECH through a small software change and it is described in the next section of this report. The APET and CPET trays were not separated into their individual streams because the software of the TITECH units at Jayplas has not been updated to allow this separation. TITECH has been able to clearly distinguish spectroscopic differences between APET and CPET and has been able to separate the two PET materials as was described in earlier in this report.

9.0 Sorting Trials to Assess the Performance on a Range of NIR Equipment

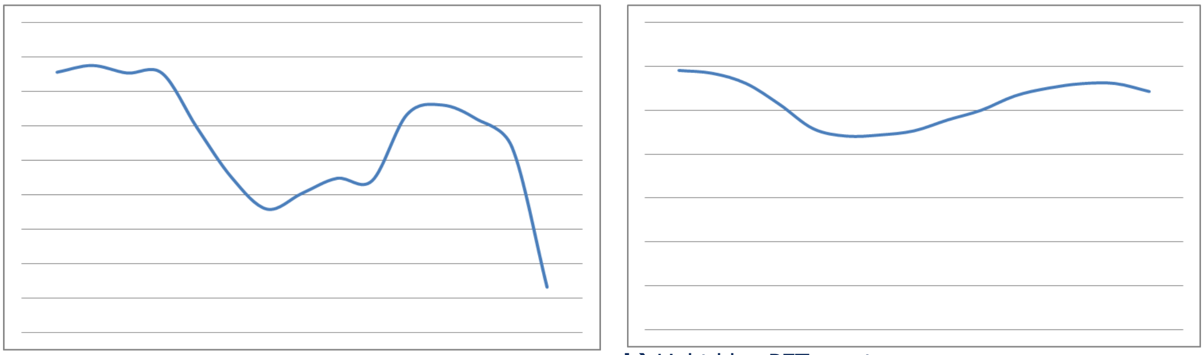
Following the large-scale trials completed at Jayplas, samples were sent to the manufacturers of the NIR and optical sorters, (TITECH, Pellenc., RTT, and S+S) and each was asked to assess the ability of its equipment to recognise the material by polymer type and to separate clear from black packaging.

For the avoidance of doubt, the intention of the work was to find colouration systems and/or technologies capable of meeting the objectives of the project. No direct comparison of individual colouration systems or technologies can be drawn from the tables and information published throughout this report. The processes in this study were tested under different conditions, using different methodologies and are therefore not directly comparable.

9.1 Separation of clear and black APET/CPET trays at TITECH

A clear transparent PET bottle on a black belt will look black to a colour detector so while it has been detected as being PET by the NIR detector the colour sensor will not identify any colour so these items are ejected into the clear bunker. By adding an NIR detectable colourant the black trays can be detected by the NIR sensor but the VIS spectrum colour sensor cannot distinguish between clear items on a black background and black items on the same background, so both clear and black packs are typically ejected into the clear bunker, as was the case during the colour separation stage during the sorting trials at Jayplas. Some of the alternative colourants used were more easily identified as a colour and so were found in the jazz bunker. TITECH commented that the spectra for clear and black products are markedly different and after sensor training and software adjustment, the new black trays were able to be easily separated from clear or light blue containers (see fig 32 for results).

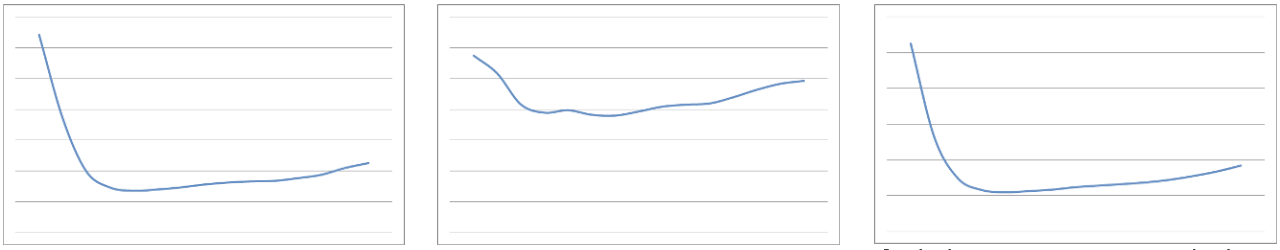
Figure 30. Spectra for Clear and Light Blue PET



a) Clear PET spectra

b) Light blue PET spectra

Figure 31. Spectra for the sample PET trays made with alternative black colourants



a) Black APET spectra - LINPAC tray ColorMatrix Dye Black-5 (0.8 % addition rate)

b) Black CPET spectra – Faerch Plast tray ColorMatrix Dye Black-5 (4% addition rate)

c) Black CPET spectra – Faerch Plast tray ColourTone Masterbatch (4% addition rate)

Figure 32. Images of sort bins after TITECH detector was optimised to distinguish black from clear PET

a) Ejected Clear PET fraction

b) Coloured PET and black trays made with detectable black colourants

9.2 Separation of clear and black Polypropylene trays at TITECH

TITECH was also asked to repeat the separation trials of black and clear with a sample of PP trays. For the test 10 black PP trays were mixed with clear and coloured PP packaging items. The aim was to eject clear PP without any black trays or other colours.

All of the black PP trays were sorted into a mixed, coloured PP / other-fraction while the clear PP was ejected except for one small clear piece, which was missed by the ejector. (See Figure 33 below).

Figure 33. Separation of black PP from clear PP

9.3 NIR testing by other manufacturers of NIR detection equipment

The purpose of these tests was to check if the different brands of sorting systems were able to identify the polymer type and the colour of each of the alternative black trays on a black background. Samples were sent to three other leading European manufacturers of NIR sorting equipment and they were asked to assess the trays for NIR polymer detectability and colour separation using their commercial sorting systems.

The samples were sent to:

- Pellenc Selective Technologies (France);
- RTT Steinert - UNISORT (Germany); and
- S+S Separation and Sorting Technology (Germany).

9.3.1 Pellenc NIR and colour identification trials

The test was performed under static imaging conditions where the objects are placed under the detector instead of on a moving conveyor and their spectral response was recorded and analysed.

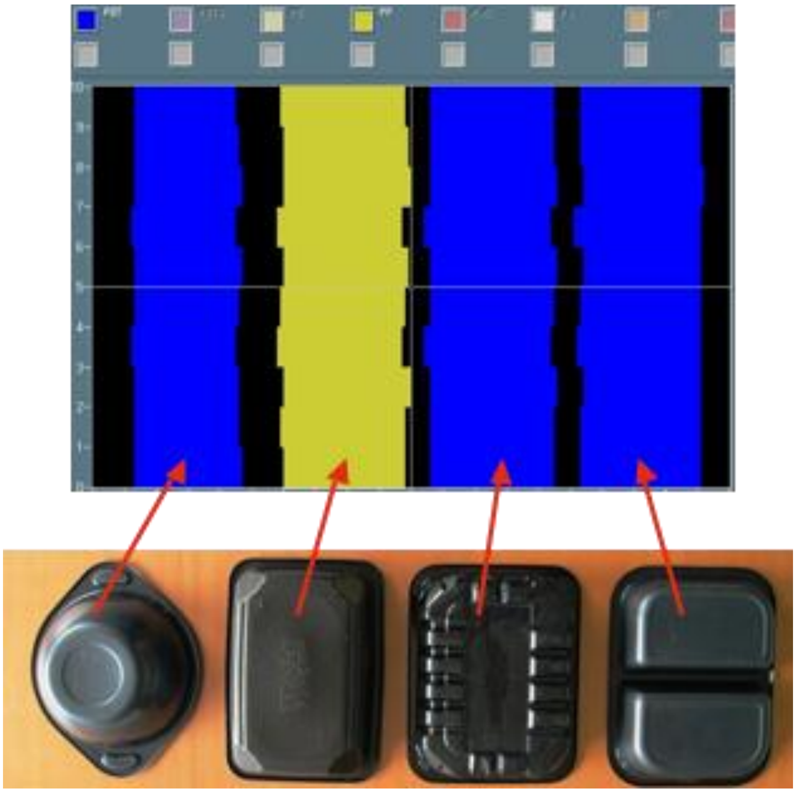
Trial Machine Configuration:

Machine type	Pellenc Mistral HR
Spectrometer:	SPIN Packaging
Colour of band:	Black

Figure 34. Images of samples sent to Pellenc for NIR and colour identification trials

Round PET 1 PP Rectangular PET 2 Rectangular PET 3

Figure 35. Results of NIR static imaging of above samples showing positive identification of PET (blue response) and PP (yellow response)



Pellenc Trial Conclusions

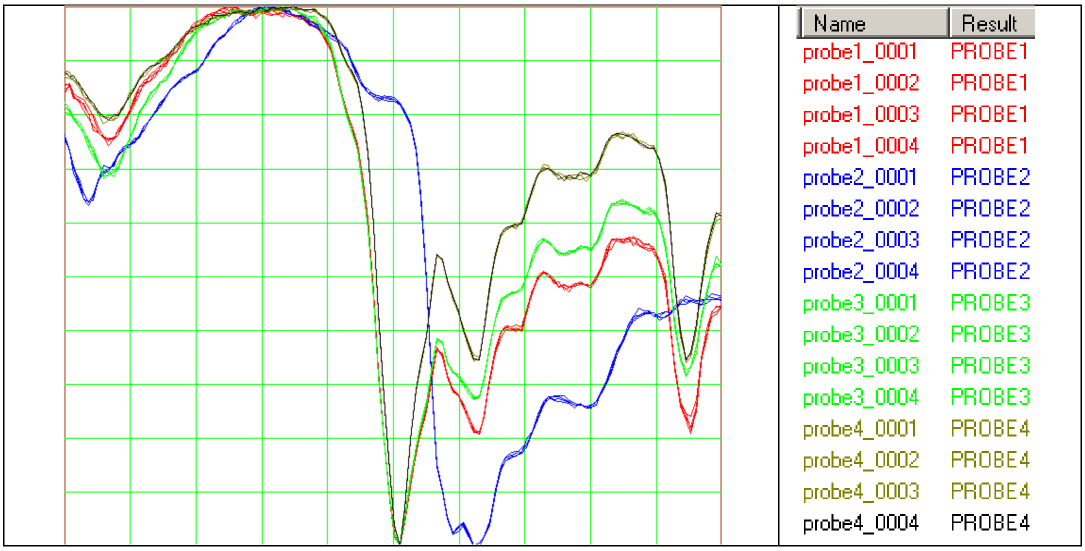
The analysis of the obtained histograms and spectra confirms that the static polymer detection of these black objects is completely feasible. Some PET trays returned a rather weak signal level under standard settings, which means that the settings must be adjusted for efficient detection in commercial operations.

9.3.2 RTT UNISORT NIR and colour identification trials

Figure 36. Images of samples sent to UNISORT

Figure 37 below shows the NIR spectra of the four tray samples. UNISORT stated that based on the NIR spectral results, its sortings equipment can detect the polymer types of all four samples (referred to as "probes"). UNISORT reported that it will be able to select and separate the tray materials from others during sorting. The results from the classification software are shown in the table on the right hand side. UNISORT also reported that all of the samples were identified as black using its colour detection camera and it can sort it by colour with a bright background or by transmitted light detection but not on a black belt.

Figure 37. NIR spectra for the tray samples using the UNISORT identification system



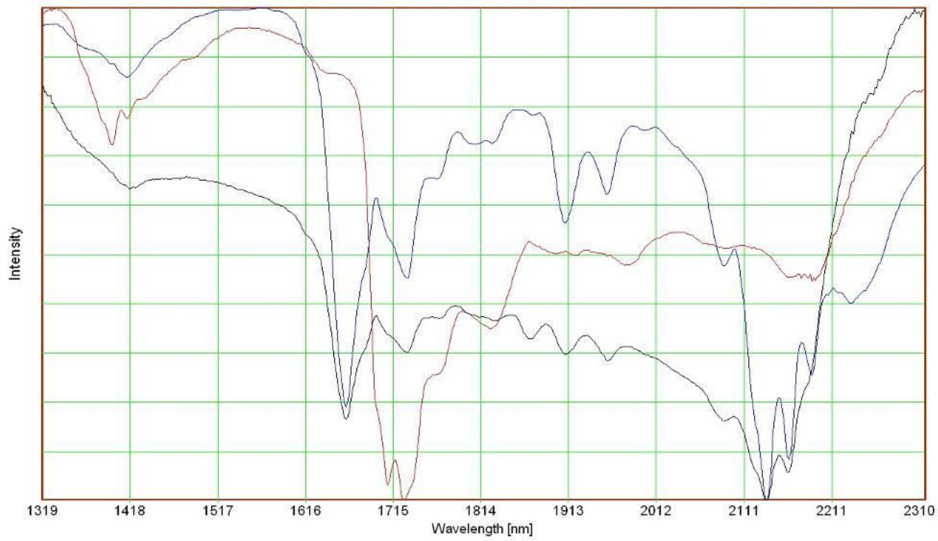
Key to NIR spectral trace above:

Probe number	Sample ID
Probe 1 (red trace)	CPET round bowl produced with ColorMatrix Dye Black 5
Probe 2 (Blue trace)	PP rectangular punnet produced with Colour Tone IRR 95530
Probe 3 (green trace)	CPET two compartment tray produced with Colour Tone IRR 95530
Probe 4 (brown / black trace)	APET rectangular tray produced with ColorMatrix Dye Black 5

9.3.3 S+S NIR and colour identification trials

S+S performed NIR polymer identification trials of the NIR visible black trays. S+S reported that the NIR spectral signals appeared quite strong and it could readily identify the PP and PET trays. It was also possible to distinguish between the APET and the CPET as they show a different signal but currently their identification routine displays them both as PET. If needed the identification file can be modified.

Figure 38. The spectra's of the different plastic types in the different wavelength areas (S+S)



S+S reported that while the differentiation for PP (red) and PET (blue and black) can be done quite easily in the lower spectra range between 1650 and 1700 nm, the classification for APET and CPET needs sorting machines which cover the extended wavelength area. S+S bottle and flake sorting systems have the extended sensor, so that clients can classify both APET and CPET where needed. Separation of the novel black PP and PET trays by colour sorting from clear was achieved by the S+S systems due to the sensor combined transmission/reflection illumination system.

9.4 Conclusions

All four manufacturers of NIR sorting machines that were sent samples of trays made with detectable black colourants found that they were able to sort the detectable black samples by polymer type and colour. The only adjustment necessary to achieve this result was to the operating software to ensure that a black tray was not misinterpreted as a clear tray against the background of a black conveyor belt. This result suggests that other manufacturers would also be able to achieve the same performance capability.

This means that the NIR detectable black colouration systems can be used with confidence in the packaging area as long as they are sorted with NIR technology with updated software.

10.0 Colour Stability of Detectable Black Colouration Systems

Samples of the trays manufactured in the large-scale trials described earlier in the report were tested for colour stability after reprocessing. Repeatedly granulating, re-melting and moulding the trays into plaques did this and test bars moulded from the three cycles were made to demonstrate the colour stability of the optimised colourants after recycling.

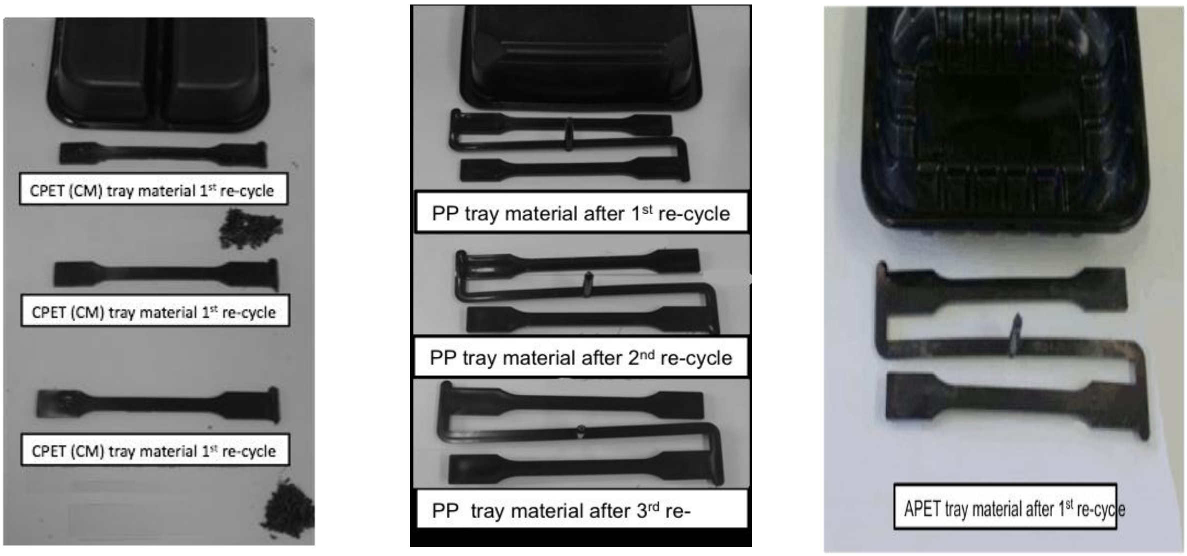
The trays were processed as individual material types and colourant combinations (APET/ ColorMatrix Dye Black 5, CPET/Colour Tone IRR 95530 and PP/Colour Tone IRR 95530) and were not mixed with each other or any other plastic materials. Firstly they were granulated using a 3mm screen then moulded into test bars and when cool examined for any changes and in particular colour changes.

Following the initial test, the test bars were then put through the granulator after thorough cleaning. The regrind was then extruded through the injection-moulding machine to produce new test bars to observe any changes in the colour. This procedure was carried out for a third time.

No significant changes were noticed during the moulding procedures and machine settings did not have to be altered from the standard setting during the three trials.

The test pieces all showed the same colour and no variation from the initial tray through to the third moulding.

Figure 39. Images of test bars after repeat re-cycles



10.1 Conclusions on colour stability

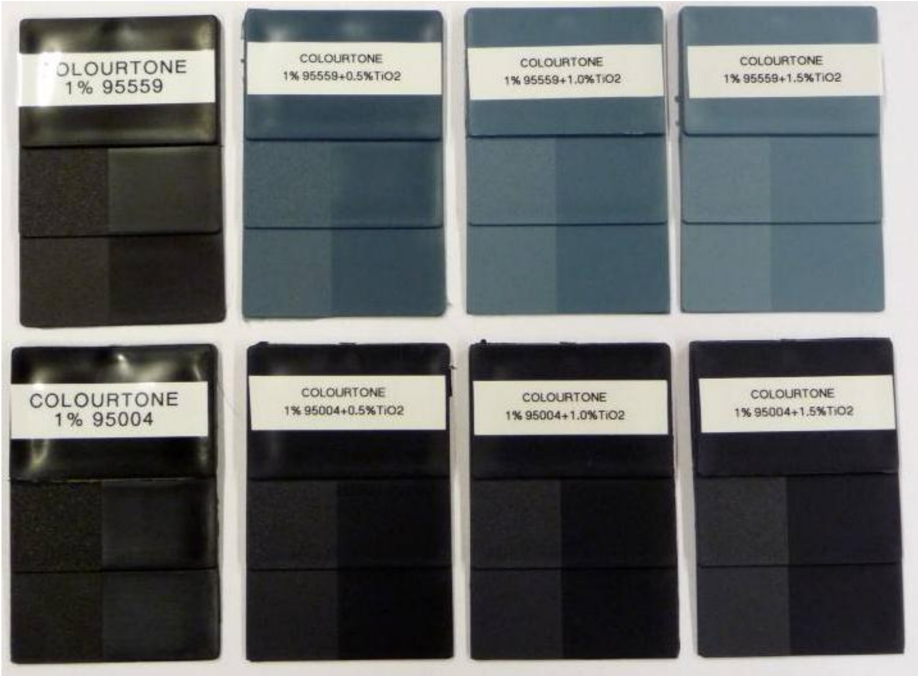
In summary, over three remoulding heat history cycles, there were no colour stability issues observed during processing of either the PP or the PET samples with either pigment. No change in colour or appearance of the material was observed once the test pieces had cooled. Therefore it is concluded that repeated recycling of PET and PP materials coloured with both the ColorMatrix Dye Black-5 or the Colour Tone IRR95530 will not impact the quality of colour.

11.0 Colour Masking Performance of Detectable Black Systems

Carbon black pigment is very effective at masking other colours and the ability to do this is helpful to plastics processors as it allows them to re-use production waste and off-cuts of all colours. It also enables reprocessors to recycle a "jazz" stream (mixed colour) from MRFs and PRFs by blending them together and recycling them into black materials. The ability of the NIR detectable black colourants to mask other colours of plastics after recycling was tested.

A range of plaques were manufactured with 1% of the improved IRR black (ref: 95559) with an increasing percentage addition of titanium dioxide (TiO_2) ranging from 0%, 0.5%, 1.0% and 1.5%. Titanium dioxide has been used to mimic a worst-case scenario of coloured plastics that could be in the 'jazz' stream and would be blended with a black colourant to make a uniform black material. The lower row, which uses a carbon black masterbatch, with the same TiO_2 content shows the superior masking properties of carbon black.

Figure 40. Comparative colour masking trial showing masking strength of alternative black colourant, Colour Tone's IRR black (95559) (top row) and the masking strength of carbon black colourant from Colour Tone (bottom row)



11.1 Conclusions on colour masking performance

While it is doubtful that such a high percentage of white would be present in a jazz fraction from a sorted mixed plastics stream and this test therefore shows a worst-case scenario of what could happen in terms of colour dilution, it is clear that the IRR colourant has relatively poor masking properties. Therefore the colours that could be included in a black fraction to be subsequently coloured black using the alternative black pigments may be limited to a recovered dark colour stream such as greens, blues and browns. Further experiments would need to be completed to identify more specifically which colours and the quantities of each that could be recycled in the black fraction.

It should be noted that while the detectable colourants do not have the same jet black masking capacity as carbon black, the final product colour and level of a jet black finish is a subjective matter. Feedback on the tray samples has indicated that some retailers have preferred the greyish tint of the "new" black packaging while others have insisted on a higher depth of colour to achieve a jet-black finish of the product. The required increase in tint strength has significant impact on the cost of the colourant and can add approximately £1 per kg or approximately 10% to the cost of masterbatch.

12.0 Environmental Impact Assessment of Detectable Black Plastics

Black plastics collected through kerbside collection systems are currently already entering UK MRFs and are ending-up in the mixed plastics packaging stream or going straight to residue and landfill as not all MRFs in the UK can process a mixed plastics stream. Information from mixed plastics recyclers such as Jayplas suggests that of the non-bottle rigid plastics stream, black packaging items make up approximately 7-10%. As collection rates of mixed plastics increase, so will the amount of black packaging within the packaging recycling chain. Materials coloured with carbon black will be destined for landfill but if they could be efficiently sorted just like other clear or coloured packaging items then they could be recycled and used to displace virgin plastics in many applications.

Implementation of either detectable black colourants, or novel sorting technologies at PRFs that enable recovery of black plastic packaging is expected to have significant net environmental benefits over the current situation where black plastics are primarily ending up in landfill.

Previous analysis by WRAP – 'LCA of Management Options for Mixed Waste Plastics 'report http://www.wrap.org.uk/document.rm?id=5497 has found that approximately 0.6 tonnes of CO_2 can be saved for every tonne of mixed plastics recycled. On the assumption that there is a minimum 30,000 tonnes per year of black packaging in the UK marketplace, and using a recycling rate of 10% for non-bottle rigid mixed plastics then recovery and recycling of 3,000 tonnes of valuable materials such as PP and PET is calculated to result in savings of approximately 1,800 tonnes per annum of CO_2 based on the assumption that the recyclate will replace virgin resins in production of industrial plastics products. If more optimistic scenarios for black plastic recovery can be achieved, then even greater benefits could be achieved.

Detailed quantitative assessments of the environmental impacts of the pigments themselves were not possible due to lack of specific data available from the pigment manufacturers. However a qualitative comparison of the stages of manufacture of the two NIR detectable colourants with carbon black is shown below in Table 8.

The comparison shows that carbon black relies on non-renewable hydrocarbon oil and is converted in a simple process that creates nano particles of carbon after thermal cracking to remove the hydrogen. The Organic Black Dye is also derived from a similar source and undergoes a more complex chemical conversion process to create the colourant molecule that is dispersed in a liquid carrier to simplify dosing into plastics later. The Colour Tone IRR black is derived from minerals that are extracted, refined, dried and ground to pigment particle sizes prior to being compounded into a polymer matrix. The processes are quite different and yet involve the common steps of mining, refining and conversion. The energy demands of each process have been estimated as being in the range moderate to low. The by-products of the manufacturing processes are simple materials for the carbon black and the IRR colourants and involve solvents for the Organic Black Dye. All of these components can be readily re-directed into other functions in modern chemical operations.

Based on an estimation of the qualitative impacts, the differences do not seem to be overly significant given the general knowledge of these processes. This means that while we cannot easily rank the colourants, we can infer that they are all going to have relatively similar environmental impacts and that the adoption of either of the two alternatives to carbon black will contribute only a small change (positive or negative) in environmental impact to the final packaging product. When compared to the overall environmental impact of the tray, the impact from the colourant in the material will be minimal as the addition rates are low 0.5-4%. The majority of the impact is related to the material type used to make the tray and its subsequent processing and product usage.

In conclusion, once the novel colourants have been converted to a solid masterbatch (M/B) or liquid colourants, they have a fairly similar environmental impact in the conversion to finished products. The benefit of being able to recycle the black packaging would be CO_2 savings of around 0.6 tonnes for each tonne of black plastic recycled. This is based on the avoidance of using virgin polymer when making new plastic items. Detailed data on the environmental impact of the pigments themselves is not available, but from a qualitative assessment of the various manufacturing processes, and considering that the addition rate of the pigments is relatively low (circa 0.5-4%) the environmental impacts of the pigments are negligible when considering the whole pack and are similar to carbon black.

Table 8. Comparison of the key manufacturing factors of two detectable colourant systems with carbon black and their environmental impacts

Factor	Carbon Black	Colourtone IRR Black Colourant System	Organic Black Dye Black	Special Comments
Raw Material Source	Petrochemical derived from oil	Refined minerals from native ore	Petrochemical derived from oil	All abundant currently
Method of Raw material extraction	Oil mining on land/under sea	Mining	Oil mining on land/under sea	Oil increasingly in difficult locations
Availability of raw materials	Widely available from oil	Widely available	Widely available from oil or coal	All abundant currently
Manufacturing Steps	Refined to Oil Cracked at high temp to carbon black. Cooled and bagged.	High temperature calcinations, particle size control and packaging	Aromatic chemicals converted to colourant materials in liquid carrier	Carbon black is efficiently made. IRR is simple to make. Organic dye needs many steps
Energy Intensity	Thermal, not considered to be intensive	Thermal, not considered to be intensive	Several steps of chemical conversion. Low to Moderate	None of the materials are high consumers of energy.
By products in manufacture	Hydrocarbons and Hydrogen	Natural gas combustion products	Solvents, and salts	Dye needs to direct residues
Issues during manufacture	Dust needs to be controlled	Dust needs to be controlled	Need to remove all starting materials from product	Dye needs purification
Toxicity of Base material for packaging.	Food grade versions are available. Must avoid the non-food grades	Food grade (pigment is non-toxic)	Food grade versions are available. Must avoid the non-food grades	All available as food grade
Application in plastics	Compounded at melt temp 200-280 °C	Compounded at melt temp 200- 280 °C	Compounded at melt temp 200- 280 °C	All are thermally stable
By products of compounding process	Dust and fines	Dust and fines	In liquid carrier format little waste	All manageable by-products
Toxicity M/B in landfill	Negligible due to encapsulation	Negligible due to encapsulation	Negligible due to encapsulation	Non-Toxic
Toxicity M/B in water	Negligible due to encapsulation	Negligible due to encapsulation	Negligible due to encapsulation	Non- Toxic
Typical dose rate	0.5 to 1.0 % w/w	0.5 to 2.0 % w/w	0.5 to 1.0 % w/w	Non- Toxic
Toxicity of Plastics in landfill	Negligible due to encapsulation	Negligible due to encapsulation	Negligible due to encapsulation	Non- Toxic
Toxicity of Plastics in water	Negligible due to encapsulation	Negligible due to encapsulation	Negligible due to encapsulation	Non- Toxic
Toxicity products in combustion	Toxicity of combustion by-products is controlled by plastic type. Carbon Black will combust to CO_2 and water	Toxicity of combustion by-products is controlled by plastic type. Will combust to CO_2 and water and residual oxides (pigment will survive combustion).	Toxicity of combustion by-products is controlled by plastic type. Will combust to CO_2 and water.	The plastics dominate this aspect.

13.0 Economic Assessment of Alternative Techniques for Black Packaging

A cost assessment was carried out in order to provide a preliminary evaluation of financial viability of the alternative black colourants compared with the carbon black pigments. This would inform discussion with the industry and establish the case for further work.

NOTE:

"WRAP has estimated the difference in tray price between carbon black and the new reflective colourant, based on pricing information provided by the colourant manufacturers in April 2011. WRAP does not have any influence over the price of the colourant, and is not suggesting these prices as Recommended Retail Prices. WRAP takes no responsibility for any increase or decrease in colourant price from these estimations, and the purchasers are responsible for securing a confirmed price themselves. These prices should not be used to try and fix a price, obtain a discount, or influence competition with any colourant manufacturer at any time in the future - the prices herein are estimated purely for the purposes of this report.

13.1 Alternative black colourants assessed

Alternative black colourants are currently more expensive than carbon black and the colourants can range in price range of (£10- £100 per kg). In general the colourants that were successful in the NIR detection trials were in the price range of (£10-£16 per kg). It should be noted that these costs have not been optimised or based on large volume orders, and in many cases they are also comparable in price to specialised colours manufactured for well- known high street branded packaging.

There were other NIR detectable black colourants and dyes that were not considered for these trials as the indicative costs from suppliers are over £100/Kg. These colourants while interesting, are currently designed for niche high-value applications and not low-cost high volume items such as plastic packaging.

13.2 Estimation of costs of alternative black colourants assessed

Carbon black colourants are low in cost. The cost of alternative black colourants will be critical to economic feasibility and commercial adoption in price sensitive markets such as plastic packaging.

Sicopal Black K 0095 and ColorMatrix Dye Black-5 costs are about 8 times the cost of conventional carbon black masterbatch and would add about 15% to the materials cost of the pack. While this is significant, optimisation of the addition rate and price reductions for high volumes are expected to make the overall cost/benefit ratio more attractive. Colour Tone pigment cost is around 4.5 times the cost of conventional carbon black.

The indicative costs provided by the pigment suppliers are provided in the table below and approximate costs have been calculated at various addition rates and compared to conventional carbon black masterbatch for two examples – a lightweight PP tray and a heavier CPET 'Ready meal' type tray. The colourant cost is shown per 1000 trays and as a percentage of the base material cost. The actual cost impact may also depend on the volume of packaging produced. Adoption of alternative black colourants in high volume packaging products is expected to result in greater production efficiencies of the components used in the formulations of alternative black colourants and their cost is expected to reduce. In some cases, additional costs may also be required for conversion of colourants in powder form to masterbatches.

Table 9. Summary of cost evaluation data

Colourant	Cost £ / Kg	Tray weight (g)	Base material	Base material cost/ 1000 trays	Black colourant cost/1000 trays @ addition rate:			% Colourant cost of base material cost @ 1%
					0.50%	0.75%	1.0%	
Sicopal K 0095	£16.20	11	PP	£10.12	£0.89	£1.34	£1.78	17.61%
		25	PET	£23.00	£2.03	£3.04	£4.05	
ColorMatrix Liquid Colour Dye Black-5	£16.00	11	PP	£10.12	£0.88	£1.32	£1.76	17.39%
		25	PET	£23.00	£2.00	£3.00	£4.00	
Colour-Tone masterbatch IRR 95559	£9.20	11	PP	£10.12	£0.51	£0.76	£1.01	10.00%
		25	PET	£23.00	£1.15	£1.73	£2.30	
Carbon black masterbatch	£2.10	11	PP	£10.12	£0.12	£0.17	£0.23	2.28%
		25	PET	£23.00	£0.26	£0.39	£0.53	

13.3 Discussion

Early investigations into the potential costs of NIR detectable black colourants clearly identified the higher cost of these colourants against the cost of commonly used carbon black colourant. However, the cost of an NIR detectable black colourant can be regarded as comparable to the cost of colourants used by brand owners and retailers for packaging that uses product specific branded colours. Special masterbatch colourants such as purple, silver or gold, which are often used in food trays, have similarly high prices when compared to the alternative blacks (see table 10 below).

Table 10. Indicative costs of coloured masterbatches per tonne

COLOUR	£ Per tonne of coloured masterbatch
Carbon Black	£2,100
Grey	£2,500
Blue	£4,000
Special Branded Purple	£7,000
Colour Tone IRR95559	£9,200
Special Branded Gold	£9,500

The major impact of using alternative colourants is the higher cost compared to conventional carbon black masterbatch. Indicative estimated cost impacts are given in table 9 and show the estimated cost differences per 1000 trays. The indicative increases in cost of using the alternative pigments developed in this project in place of carbon black, is in the region of £1.80 to £3.50 per 1000 PET trays, at a 1% addition rate. For thinner PP trays this would be in the region of £0.75 to £1.50 per 1000 trays at a 1% addition rate. That's around 0.075 pence to 0.35 pence per tray at a 1% addition rate, but comparable to some other colours. At this addition rate, this implies an incremental cost per tonne of trays manufactured in the region of £70-£140.

However it should be noted that these indicative cost ranges are based on preliminary prices and it can be expected that if the supply chain wishes to implement alternative black pigments that commercial prices would be negotiated on the basis of large volumes, and therefore could be significantly lower.

Results from trials indicate that switching from a 1% carbon black addition rate to one of the new colourants may require an increase in addition rate to get the same depth of colour. A higher addition rate may also be required during reprocessing for improved masking capability. The cost comparisons for new product development need to consider the level of addition rate and this would need to be evaluated for each specific product as different addition rates may be needed depending on the product's wall thickness, etc. The cost differential will therefore be strongly influenced by the final addition rate and what is an acceptable black finish.

It should be noted that while the detectable colourants do not have the same covering power as carbon black, the final product colour is a matter of subjectivity. Some retailers have preferred a greyish tint to the packaging while others have insisted on a higher depth of colour close to a jet black finish. Improvements in re-formulations of colourants will continue to improve tint strength, which may mean lower addition rates but these changes can also significantly add to the cost of the colourant and some suppliers have reported added costs of approximately £1 per kg or approximately 10% to the cost of masterbatch. Adjusting the tint is therefore an option that could be explored by retailers and packaging manufacturers based on their desire to control costs and adjust the colour for customer appeal.

14.0 Implications of the Commercialisation of NIR Detectable Black Plastics Packaging

With the introduction of black colourants that enable the polymer and colour of the pack to be detected by optical sorters currently in use at plastic recycling facilities, the recovery of black plastics packaging will become possible and typically the packaging will be collected as part of either the coloured fraction of the specific polymer stream or as part of the mixed plastics fraction.

The initiation of recovery of these plastics will mean that the additional materials would boost the economic efficiency of recycling operations because of improved material yields, lower levels of residue wastes to landfill and opportunity for greater revenues from sale of recycled resins produced from the recovered black plastics. The actual value created would depend on the ability to efficiently sort the black plastic waste products and reprocess into recycled resins that can be used in applications where the recycled black plastic resins could replace virgin plastics.

The key polymers used for black plastic packaging are PP, CPET and APET (making up around 80% of the market) with, HDPE, PS and PVC making up the remainder. The recycling and reprocessing issues associated with each polymer are quite different once they are separated into their specific coloured plastic fraction rather than being left in the residue or as mixed plastic bales for sale. Markets for recycled black PET and PP are also quite different but both materials are valuable materials and can be used in many different applications. For PET, there are potential end markets for APET and CPET combined, as well as for separate APET and CPET streams.

APET: The end markets for coloured APET are typically fibre and strapping. The fibre market uses APET for either textile that could be coloured dark shades such as black or as fibre for roofing membranes where it is covered with bitumen. In both cases the presence of the black plastics is not a hindrance to the value and market acceptance of this stream of packaging. In the case of strapping the predominant colour used in the market is green (achieved via masterbatch addition) which permits the majority of coloured APET bottles (blue and green) to be used. The presence of other colours will potentially shift the shade of green depending on the range of colours in the coloured feedstock, their tint strength and their abundance. It has been noted that the detectable colourants have a relatively low tint strength and initially the abundance would be quite low compared to the presence of green and blue bottles and would be comparable to the impact of brown and red PET bottles. In this case no action is needed however it would be useful to understand the critical levels of detectable black colourant on the shade shift, the level of masterbatch needed to compensate for the shift in colour and the influence on mechanical properties. As the abundance of the detectable black trays increases in the waste stream through increased recycling levels, there are a number of potential market options.

Firstly to look at the economics of colour separating and recycling the black separately for reuse into sheet that will be made intentionally black with consequent savings in masterbatch, especially if the trays were to be detectable once again.

Secondly to examine the possibility of developing a specific rPET designated black strapping product that may have economic advantages for the strapping companies.

Thirdly the black APET stream (and even other colours) could be combined with the black CPET stream for reuse in the food grade black CPET sheet or black textile fibres that would benefit from the nucleating agents in the CPET. The last two markets would need to be supported by additional development work.

CPET: It has been demonstrated in this study that CPET and APET can be separated using NIR allowing a relatively pure stream of CPET to be recovered since the only recovered CPET trays would be detectable black

CPET trays. The presence of any detectable black APET would be a positive increase in the volume of this fraction. This would allow the closing of the loop on CPET trays back to food grade for the very first time and allow this very significant volume of materials to be recovered from the waste stream. At lower recovery rates the CPET trays could be directed into the coloured APET fraction for specific use in applications such as fibre or strapping, where CPET would not cause problems such as it would in the bottle or sheet market. The same caveats as discussed for APET would apply.

APET multilayer trays: APET trays are often made with a PE top layer that is used for easy bonding of lidding films for product containment or for controlled/modified atmosphere conditions. This is practised in both black and other coloured trays. The inclusion of the detectable black trays with the PE layer into the coloured APET stream will mean that these materials will be destined to be sold into the fibre and strapping markets or potentially the CPET market if it is sorted deliberately into that fraction. In each case the presence of PE will act as a nucleating agent causing the material to behave like a crystallised material such as CPET. This is therefore useful in CPET but not in APET. However there is always a problem in the reprocessing of these laminates due to the melting of the PE during the drying phase required for PET recycling, as the PE melts at a lower temperature than the drying temperature used for PET. This issue could be eliminated by the use of modified grade of PET that provides much easier bonding to the tray surface but do not create the process stickiness problem caused by the melting of PE. A focused R&D project on this topic could investigate this issue and potentially simplify the in-plant and external recycling operations. Until this issue has been resolved it may be sensible to restrain using the detectable black colourants in these products to avoid any complications with the recycling of coloured PET.

PP: The black PP stream represents the most significant volume amongst the black plastics packaging and its recovery would boost the volume of the coloured PP fraction. Natural PP from ice-cream containers, cream pots and take-away containers exist in the stream, but feedback from recyclers such as Jayplas suggests that recovery levels are currently low and for this reason PP from waste streams is currently only sorted by polymer type and then reprocessed together with other colours. At low recovery rates nothing needs to be done since this fraction is usually coloured black, but as recovery of PP increases, it is likely to be sorted into a natural stream and a coloured stream. Of course the ideal outcome would be to use the detectable black colourants in preference to carbon black during reprocessing, however the cost differential and the market price for recycled PP would usually result in carbon black being chosen by the reprocessor. If the end markets were in durable and long-lived products, then this would be an acceptable outcome, as the recovered PP would be displacing virgin PP and would only re-enter the waste stream after long periods of time, but for fast moving consumer packaged goods that enter the waste stream in large volumes and quickly after use, the use of detectable black colourants would help improve closed loop recycling. At higher recovery volumes of detectable black PP, the separate recycling of this fraction could be achieved using newly developed food grade PP recycling processes.

HDPE: The black HDPE stream is a minor fraction in the coloured HDPE stream that is full of many strongly pigmented colours and the first option would be to allow the material to be recycled along with these materials in most cases with a consequential increase in recycling efficiency. However there have already been expressions from multi-national brand owners that currently make black containers to have these recovered and recycled back into the same product as is practiced with other colours such as blue, pink, white, yellow and red. The development of these recovery loops may well provide the basis for increased recovery due to increased market demand for products to contain recycled content.

PS: The recovery of black PS in packaging could lead to a boost to the recovery rates of PS since little PS is currently recovered from the PS packaging waste stream. It is expected that these materials would be processed with the coloured PS fraction due to the low volumes of PS in the mixed plastics waste stream. There could however be very significant opportunities for the use of detectable black plastics in the electrical and electronics sector as the mandatory recovery targets could well support the adoption of these more expensive colouration systems. Spectroscopically detectable black colourants specially formulated for styrenics such as PS/HIPS/ABS, which are widely used in electronic goods could be adopted and this would assist in the economic recovery and separation of these polymers at the end of the products life cycle.

PVC: The volume of PVC packaging has been steadily declining and the inclusion of black PVC into the coloured PVC stream should not cause difficulties as this mixture is currently pigmented black into many other long term and durable products such as shoe soles, hose pipe inners, flooring tiles and backing for floor coverings.

15.0 Recommended Next Steps

Where possible, WRAP would recommend the use of alternative colours to black so that the pack can be easily detected and recycled. However, where a black colour is desired, in order for the packaging supply chain to more widely adopt the new colouration systems, further optimisation, testing and validation work is required. This covers issues relating to the economics, manufacture, filling, shelf stability, distribution and sorting during recycling.

15.1 Further investigations to identify which colours & quantities can be recycled together

Masking strength is very important for packaging convertors and reprocessors, and further investigation is required to understand in greater detail what the implications are regarding the lower masking strength of the alternative pigments, and how this could be either accommodated or whether the pigments could be further optimised to improve their masking capabilities. A series of trials to identify the colours and quantities of specific colours that impact the ability to mask a mix of coloured products during recycling would provide important information. Further optimisation of the pigments may be required to provide a higher level of covering power.

15.2 Large scale demonstration trials

Once the colourants are shown to provide the required level of masking strength, then retailers and brand owners could work with their suppliers to carry out large scale trial production runs on specific packaging. Once shown to be successful, the range of products using the colouration systems could be widened to eventually replace the carbon black systems.

This may be in conjunction with further technical optimisation of the colouration systems on the specific polymers and packaging systems which would have the potential to improve performance and reduce costs through improvements in the scale of supply of specific masterbatches.

Large scale production trials on specific packaging would involve assessments across the whole supply chain, including tray manufacture, filling and sealing production trials, shelf life trials, microbiological and organoleptic testing, in store and distribution trials, and economic assessments. If rolled out, communications to update NIR users on modification of NIR software settings to enable colour sorting of black packs would be required.

15.2.1 Colour optimisation, migration, and bleed tests

Colour shades and tint requirements are subjective and so optimisation based on customers aesthetic requirements is an important step.

Any new developments for ready meal products would need to include stringent colour migration and bleed tests because of the very specific need to ensure that colourants or components of the colourants cannot migrate into the food when the food is being re-heated or cooked in the tray.

For CPET, Colour Tone's IRR95530 was not suitable for use in CPET ready meal products. Colour Tone developed an improved colourant (IRR95559), by removing one of the components found to be bleeding and replacing it with a stable component that resulted in the optimised colourant being suitable for use in PP, APET and CPET packaging including oven ready applications. Whilst the bleeding/colour migration property of the reformulated pigment was tested in plaque form, a sample tray was not produced and would need to be completed to ensure that this pigment looks acceptable when thermoformed into a tray. Further optimisation may be required to ensure this is aesthetically acceptable in tray form.

The colourants used are approved for use with food. However suppliers need to ensure that the colourants are food contact compliant under ambient storage conditions as well as under defined conditions for specific applications such as cooking or re-heating of ready meals. Therefore migration tests will need to be completed for specific food applications.

Packaging samples would need to be tested for overall and specific migration following the methods laid out in Commission Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food (as amended). Packaging would need to meet all existing performance and subjective specifications agreed between the manufacturer, the packer / filler and the retailer or brand owner.

15.2.2 Optimising hot seal and film lidding conditions

Continuous sealing trials need to be performed on production equipment. Preliminary assessments have shown that trays containing the novel black colourants developed in this project can be sealed with lidding films, but this needs to be assessed in large scale trials. Work needs to be performed to develop specific sealing, lidding and wrapping solutions for PP, APET and CPET trays.

15.2.3 Modifications at plastics sorting facilities

The sorting of the NIR detectable black packaging at the major plastics PRF's should be monitored to ensure that the correct adjustments were made to the software used by the NIR sorting equipment.
The production of a technical bulletin for businesses using automatic NIR sorting equipment would be helpful in ensuring that the capacity to recover these materials is maximised.

16.0 Conclusions

The research and development performed in this project has demonstrated that it is technically feasible to use black colourants that enable the polymer and colour to be detected using the NIR optical sorting systems currently in place at recyclers. However further testing and development work is required in order to use these colourants for specific food contact packaging applications.

For the avoidance of doubt, the intention of the work was to find colouration systems and/or technologies capable of meeting the objectives of the project. No direct comparison of individual colouration systems or technologies can be drawn from the tables and information published throughout this report. The processes in this study were tested under different conditions, using different methodologies and are therefore not directly comparable.

The project has shown that there are at least two potential approaches to enable black plastic packaging to be recyclable; the use of NIR detectable pigments in conjunction with conventional NIR automatic sorting equipment and the use of MIR automatic sorting equipment at MRFs, PRFs and reprocessors.

A range of pigments and dyes that provide a black appearance to the pack yet do not absorb the NIR signal used to detect and classify the type of plastic appear close to being able to be implemented subject to the further work discussed. The development of MIR automatic sorting equipment is still emerging and not available on a commercial scale in the waste recycling sector yet. However this technique may provide an important sorting technique in the future.

The use of detectable black colourants has been shown to be effective in PP, APET, CPET, PS and PVC and most likely, on this basis, in many other plastics. This presents opportunities for identification and therefore possible recovery and recycling of black plastics commonly used in other markets such as electrical and electronic equipment as well as automotive and building/construction industries. Given the popularity of black colour in electrical and electronic products, there is significant potential to help improve recovery rate, efficiency and throughput rate of businesses recovering WEEE and automotive plastic products.

A cost assessment was carried out in order to provide a preliminary evaluation of financial viability of the alternative black pigments compared with the carbon black pigments. This would inform discussion with the industry and establish the case for further work. The detectable black colourants are more expensive than conventional carbon black pigments with an indicative price range similar to other specialised organic pigments of the order of £9 to £16 per kg.

The indicative increases in cost of using these alternative pigments in place of carbon black, is in the region of £1.80 to £3.50 per 1000 PET trays, at a 1% addition rate. For thinner PP trays this would be in the region of £0.75 to £1.50 per 1000 trays at a 1% addition rate. That's around 0.075 pence to 0.35 pence per tray at a 1% addition rate, but comparable to some other colours. This implies an incremental cost per tonne of trays manufactured in the region of £70-£140.

However it should be noted that these indicative cost ranges are based on preliminary prices and it can be expected that if the supply chain wishes to implement alternative black pigments that commercial prices would be negotiated on the basis of large volumes, and therefore could be significantly lower.

The cost differential is heavily dependent on the addition rate required and the tint strength needed for a specific pack. The black finish requirement of a specific pack needs to be evaluated by the customer on case-by-case basis as the black finish and cost is directly related to the addition rate of the colourant.

The detectable black colourants have been tested and found to be heat stable to 300°C and recyclable without visual impairment to the appearance of the plastic products and the detectable black colourants developed are approved for use in food grade packaging.   One detectable black colourant used in CPET was also shown to be resistant to the conditions used in cooking oily foods after the formulation had been appropriately adjusted.

Packaging technologists at several UK grocery retailers have reviewed the colours of the detectable black colourants and have found them to be acceptable colours for use in packaging but further approvals by product category managers may be needed for specific product lines. Large scale manufacturing trials by three tray manufacturers proved that these colourants perform and process as well as carbon black in PP and PET sheet and thermoforming operations. The plastics packaging made with detectable black colourants was processed by addition as masterbatch and liquid colourant to plastics by extrusion, injection moulding and thermoforming without any changes to the temperatures, processing speeds and energy input used for conventional black products.

A range of manufacturers of NIR automatic sorting equipment has tested the black trays made with detectable black colourants and all have successfully identified the polymer types and the colour of the sample trays. It is expected that this is a general principle that will apply to other NIR sorting machines.

The environmental benefit of using the detectable black colourants in plastics could generate carbon savings of around 0.6 tonnes of CO_2 per tonne of plastic recovered and used in place of virgin plastic to manufacture new items. The overall environmental impact of the colourants is expected to be low due to the low rate of addition (0.5-4%) and has been estimated to be very similar to that of using carbon black colourants based on the current knowledge of the two colourant systems.

The detectable black colourants used in this project have allowed the identification and separation of black APET and black CPET for the first time. This is because CPET is almost exclusively used for black ready meal tray packaging and until now black CPET packaging trays containing carbon black could not be detected. This development foreshadows the opportunity to recycle both types of materials back in to new plastics and fibre applications including a closed loop for food packaging.

The use of detectable black colourants will allow operators of MRFs and PRFs to obtain higher yields from the inclusion of black plastics from a stream of packaging that has been estimated to be in the region of 30- 60,000 tonnes in the UK. This would deliver economic benefits on the form of associated revenues and avoided landfill costs to the recycling industry and these economic benefits would grow proportionately as the volume of mixed plastics collected for recycling increases. NIR detectable colourants also present potential opportunities across a number of other industries where it would be desirable to recover and recycle black items, such as electrical and electronic equipment (WEEE) as well as automotive and building/construction industries. These recycling operations would benefit from the increased recovery rates and higher throughputs that could be achieved with NIR sorting equipment.

In summary, this research has shown that a range of black colourants are now available to the plastics packaging industry and other sectors (including EEE and automotive) that would allow the automatic detection and recovery of these materials so that they can be used in new applications as substitutes for virgin plastics with subsequent improvements in recycling revenues as well as savings in carbon emissions and landfill volumes.

Appendix 1: Review of Potential Approaches to Sorting Black Plastics

This project explored four possible approaches to enable automated sorting of black plastic packaging. These were: alternative spectroscopic techniques, physical sorting methods, the development of alternative colourants and the addition of detectable markers and are discussed in detail here.

Use of alternative spectroscopic techniques without changing colourant types

Mid-infrared (MIR) spectroscopy for polymer identification is well known, but suffers from two deficiencies for automated sorting. The time of detection is at least in the order of 1 second, which is slower than with NIR and the computational requirements to match the product spectrum to reference spectra are also more complex due to the large number of peaks in the mid infrared 'fingerprint-region'. However, absorption by carbon black is less strong in the mid-infrared region than for either the NIR or visible regions (Murphy 2001). MIR is recommended for identification of black plastics for this reason, although there are difficulties for industrial applications around lack of robustness of the equipment due to the need for close proximity of the sample and detector (Fisher 2003), and sensitivity to the surface characteristics of the articles to be sorted (Eisenreich & Rohe 2000). MIR has found use in identification of materials in automotive parts, but does not appear to be viable for use in high-speed automated sorting of post-consumer packaging without significant equipment development.

Raman spectroscopy is a potential alternative to NIR that has received considerable attention in plastics sorting. It is an emission technique that does not rely on the measurement of absorbed or reflected radiation. A laser operating in the near infrared or visible region is used to excite the material, producing a characteristic emission spectrum in the infrared region through the Raman scattering effect. The potential of Raman spectroscopy for plastics identification in recycling operations has been long known (Florestan et al. 1994) but as with NIR spectroscopy, black pigmented samples cannot normally be measured, due to high surface absorption of the incident laser pulse and extra fluorescence from the carbon black. Recent advances have been made to solve this problem however this equipment is currently targeted for automotive and electronic waste rather than sorting of post-consumer packaging. Sony claims its advances make Raman spectroscopy well suited to plastics sorting, including black plastics (Sony Int Europe GmbH, 1999). Raman spectroscopy is a potential method for sorting black post-consumer packaging, subject to a commercially viable detection system becoming available.

The Unisensor POWERSORT 200 flake sorting system uses Laser Raman Fluorescence Spectroscopy and is currently being used to sort black plastic flakes in the automotive industry (http://www.unisensor.de/). The system can sort black flakes but is not designed to handle whole packaging items such as black pots, tubs or trays.

X-ray fluorescence (XRF) spectroscopy is a technique that is already used commercially to detect and sort PVC containers. However, it is difficult to quantify elements of low atomic number by this technique. PE, PP, PET and most other common polymers contain only the low atomic number elements (H, C and O) and cannot be differentiated using XRF.

Laser-induced breakdown (laser-induced plasma) spectroscopy (LIBS / LIPS) technique could be suitable for on-line, real time detection having rapid response time and the ability to detect spectra remotely. The sampling time for the generated emissions from the laser heated sample is very short as plasma is generated within nano-seconds (ns) of the laser pulse. The emissions are collected and analysed then the detecting channel is cleared for the next sample. Identification and calculation time are believed to be short, but the overall response time is not known, as there are no commercial systems available yet. Pigmentation of the product will not stop the production of the plasma or interfere with the emission detection. Hence, black articles could be identified as well as transparent or light coloured articles. Surface coatings and labels will likely affect the results, as it is only the sample surface that is measured. LIBS / LIPS is not yet a commercial technology for sorting plastics, but it shows some promise in this application.

Photoacoustic spectroscopy involves measuring the acoustic emission after laser irradiation. The laser radiation is pulsed at an audible frequency, resulting in a sound proportional to the intensity of radiation absorption (Gregoriou et al. 2000). The colour and surface condition of the article will not affect the photoacoustic response. It has been tested in recycling plants for automotive and electronic waste sorting (AMES 2002). It is not known if the robustness and response time of the technique is suitable for use in recycling plants for post-consumer packaging.

Sorting by physical methods not involving spectroscopy

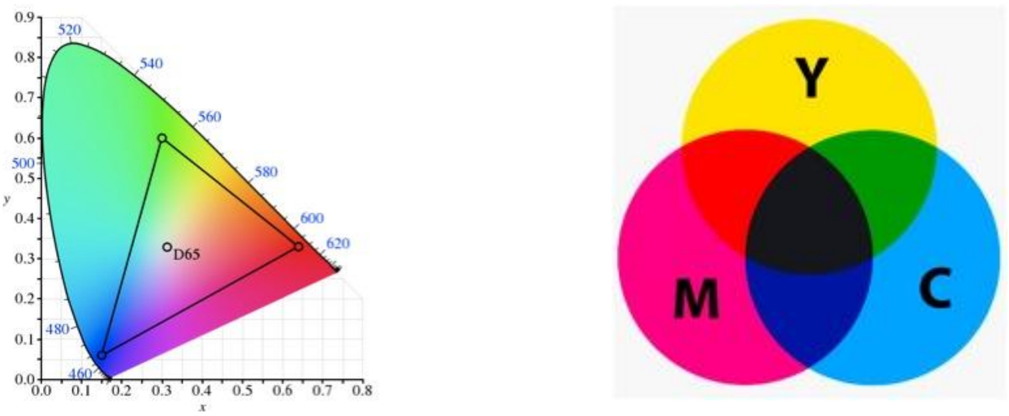
The **triboelectric charging** of a packaging item as it is rubbed against a charging material is measured through a probe, and suggested to be suitable for use in automated sorting. The tribolectric principal is only able to do a binary sort between two polymers with a positive and negative charge relative to the reference material, however a series of sorts could be utilised with different reference probes in order to separate the different plastic types based on their position in the charging sequence. Such a set-up could operate along a conveyor belt, although controlled humidity is probably required. However the charge decay time reported required to differentiate insulating plastics was substantial, in the order of 10 seconds or so, which may limit throughput. High levels of surface contamination, surface moisture or full-body labels and coatings were admitted as problematic for this technology. The separation of mixed plastics in flake form using triboelectric principles in a commercially available Hamos sorter, obtained poor results in an earlier study (WRAP 2008).

In **tack separation**, plastic articles are heated by IR radiation. Different plastics become tacky at different temperatures as they approach their melting point. This can allow low melting point plastics to be separated by their adherence to a conveyor or drum. The process has been demonstrated by a US automotive recycling company however, in practice, it is more effective to control the heating rate by using infrared radiation at wavelengths tuned to the main NIR absorption band of the plastic to be separated. This system would be less effective with conventional black plastics because the selective NIR absorption is masked by the carbon black absorption.

Development of an alternative NIR detectable black colourant

A black pigment can result from a combination of several pigments that collectively absorb all colours. If appropriate proportions of three primary pigments are mixed, the result reflects so little light as to be called "black". Black can therefore be described as a lack of all colours of light, or a combination of multiple colours of pigment. Mixing yellow and cyan produces green colours; mixing yellow with magenta produces reds, and mixing magenta with cyan produces blues. In theory, mixing equal amounts of all three pigments should produce grey, resulting in black when all three are applied in sufficient density, but in practice they tend to produce muddy brown colours, or lighter shades of black.

Figure 41. Example of the RGB Colour Index and an example of how black colour can be achieved by mixing the key colours of magenta, yellow and cyan together (Source: The International Commission on Illumination)



It may be possible to select black colourants that absorb in the visible region, but reflect or transmit NIR radiation, allowing for the standard NIR detectors in automated sorting to register a useful spectrum from a black packaging article. However, the IR absorptive properties of standard carbon black colourants can be a benefit when thermoforming trays from plastic sheet because the higher infrared absorption increases the operating speed of the thermoforming equipment by enabling faster heat up of the sheet to moulding temperature.

NIR transparent colourants

Black colourants have been developed for use in coatings, paints and resins that act to reduce solar thermal gain in dark coloured building or automotive products by either being low in absorption, or reflective of infrared radiation. These colourants may prove to be suitable for black colouration in plastic packaging, yet enable detection by NIR spectroscopy. NIR transparent black colourants are used for laser welding plastic components. They allow infrared radiation to penetrate through one component to reach the interface with a second component that contains IR absorbers. The IR radiation causes local heating at the interface allowing the two components to be welded together. Examples of black colourants for these applications are the perylene colourants (BASF et al. 2005). These are organic perylene colourants that do not absorb strongly in the infrared region, but can be produced with high opacity and blackness.

A number of black colourants with high transparency in the NIR region were identified in the literature review:

- BASF Black NIR transparent colourants, such as Lumogen Black FK4280 and FK4281;
- Fuji Xerox developed colourants with high black colouration and NIR transparency for photocopy toner; and
- Treffert GmbH (www.treffert.org) produce specialised masterbatches for laser welding based on soluble dyes.

The BASF literature on Lumogen colourant shows significant transmission at wavelengths longer than 1000 nm. This is almost halfway in the near infrared spectrum (700nm-1400nm) as is defined by the International Commission on Illumination. The Fuji Xerox patent application notes that they could also be used for laser welding of plastics (Hasegawa et al. 2009). They are based on a squarylium compound combined with phthalocyanine blue colourant. Treffert report that that have both laser absorbing and laser transparent types (Glaser 2006).

NIR reflective colourants

Paints and coatings with high infrared reflectivity or low absorption are now well known. They are useful for applications such as coatings for roofing tiles that enable dark colours to be used with less heat gain than normally achieved, and housings for lighting systems to reduce heat build-up.

Several commercial examples exist of IR reflecting black colourants that can be used in plastics, such as Sicopal Black K 0095 from BASF and Black 10P922 from Shepherd Colour Company. An IR reflective colourant at low concentration in the polymer matrix could allow for a useful infrared spectrum to be obtained in reflectance mode.

Markers that provide an identification signal

Fluorescent Additives

Eastman (www.eastman.com) has worked on molecular markers that would be incorporated into the polymer resins during polymerization (Cushman et al. 1996). These molecules can be copolymerised into PET and fluoresce in the NIR region when illuminated with a laser. They can be selected from chemicals that do not absorb visible light, and so do not affect colour and clarity of the base polymer, at least at the low addition levels required. In principle these markers could be added during production of the base polymer, but this would require international agreement between plastics suppliers. A more practical approach would be to add them at the convertor stage for specific products, such as black trays, by adding in a masterbatch.

The use of fluorescent markers for sorting post-consumer packaging has been investigated at Cranfield University (Ahmad 2000). The markers could be detected at concentrations between 0.5 and 20 ppm with optical sensors. Combinations of four different fluorescent markers were shown to provide a basis for an automated sorting system. However, colourants in the plastics reduced the fluorescent signal yield and with carbon black colourants, identification became impossible. Carbon black colourants will attenuate the excitation radiation and the fluorescent response in either the UV, visible or NIR spectra. Hence the feasibility of this approach will depend on the addition rate required for effective detection in black materials. Alternatively, they could also be incorporated in coatings printed onto black trays. This would avoid absorption by the black colourant but would require an additional secondary process. The primary difficulty would simply be getting sufficient compliance from the large number of packaging suppliers.

Taggants

Taggants are a more sophisticated application of fluorescent additives. They are generally in a fine particulate form with a combination of colours that encode the identification information. This is detectable as a specific response when exposed to particular frequencies of radiation. They are aimed at brand verification, anti-counterfeiting and traceability applications by offering a large number of unique identification codes. Taggant technologies suitable for addition to plastics have been developed by a number of companies.

Microtrace (www.microtracesolutions.com) employ a range of technologies to provide a numeric code sequence from colour-layered particles. A UV laser is used to locate the particles by exciting a visible green fluorescent response. A special detection unit then uses IR frequencies to read the particle's code. The effectiveness of the system in black plastics would be limited by absorption of the IR and it is intended for manual verification applications rather than automated detection. Because individual particles can be located, allowing close positioning of the detector, very low addition rates in the range of 0.035% – 0.075% can be used at a cost of a few pence per kg of plastic. The technology is not expected to be suitable for plastics sorting.

Addmaster (www.verimaster.co.uk) has a system based on optical detection technology with special detector systems. Taggants are available with 6 colours and are presumably combined to provide a unique fluorescent signature when excited by laser light. 'Invisible' taggants with no visible light emission are also available. Sorting of materials for closed loop recycling systems is listed as an application. Addmaster has investigated addition to dark coloured and black materials and found that very high loadings, of 15% or more, were required for detection and this would clearly be uneconomic. High-speed detector systems are not currently available and would need to be developed.

3M Light Reveal Authentication products emit visible light frequencies when excited by a laser (3M 2009). A similar system, the Kodak Traceless system uses proprietary markers, which can be integrated into plastics and inks, and are detectable with special readers (Swedberg 2008).

DNA taggants have been developed for high security verification applications (www.dnatechnologies.com). DNA gene segments contain long sequences of four nucleotides, which can be used to provide a complex code. The DNA segments can be incorporated in inks and printed onto plastic articles. They are identifiable by using biochemical laboratory procedures to replicate the DNA and read the sequence. This would not be practical for high-speed automated sorting.

As with fluorescent dyes, the effectiveness of taggants will be reduced by absorption from black colourants, but may be effective in a coating or label added to the package.

RFID

Radio-frequency identification (RFID) tags are widely used for tracking plastic pallets, mobile garbage bins and reusable crates. Passive RFID tags derive power from incident radio frequency (RF) radiation and do not require a battery. The main components are a copper aerial loop, an integrated circuit chip and plastic encapsulation. They can be less than 1 mm in size and weigh just 0.25 g, in the form of a flat square, which can be embedded in a plastic article during moulding, or adhered to its surface. RFID tags emit an RF identification signal when they detect RF radiation at the correct frequency and intensity. Carbon black colourants would not absorb RF radiation very much. The cost and size of RFID tags has reduced to a point where tagging of plastic bottles and pallet shrink-wrap for recycling has been proposed (Stigall 2006).

There is a significant risk that widespread use of RFID tags would compromise recycling by contaminating the recyclate when granulated, and are normally not recommended for this reason. Typically, they contain 40% metals as well as thermoset plastics. Removal during recycling may be possible, for example with a soluble adhesive and a wash stage before granulation, but their use adds additional process complexity.

A related concept would be to use lower-cost 'chipless' RFIDs (RFID Journal 2009). These can be made from an electrically conductive polymer that could be printed onto the packaging. This latter approach may present solutions for packaging in the future.

Conclusions

Of these four approaches, alternative spectroscopic techniques and alternative colourants were selected for further investigation and developed further in this project. However, only the alternative colourant technique would allow sorting of black trays with existing NIR based mixed plastics sorting facilities. All of the other techniques would require investment in additional sorting equipment, ranging from an additional spectroscopic sorting stage through to reconfiguration of the plant to allow physical sorting of the out-throw stream.

Appendix 2: Review of spectroscopic technologies for detection of black plastics

Spectroscopic tests were performed on commercial samples of black tubs and trays that are currently being used in the UK retail marketplace. The tests were performed to ascertain the detection response of a variety of black plastic packaging with standard NIR sorting equipment. Alternative spectroscopic techniques such as MIR and Raman Laser spectroscopy were tested to determine their potential for sorting commercially available black plastic packaging that contains carbon black pigment.

The spectroscopic technologies used for material detection and assessment of commercially available black tray packaging in UK retail stores included:

- Near Infrared (NIR) Spectroscopy using a TITECH Autosort System;
- Mid Infrared (MIR) Spectroscopy using a laboratory testing facility with a static MIR spectrometer;
- Laser Raman Fluorescence Spectroscopy tests using a UNISENSOR Powersort 200 System; and
- Near infrared (NIR) Technology & Testing.

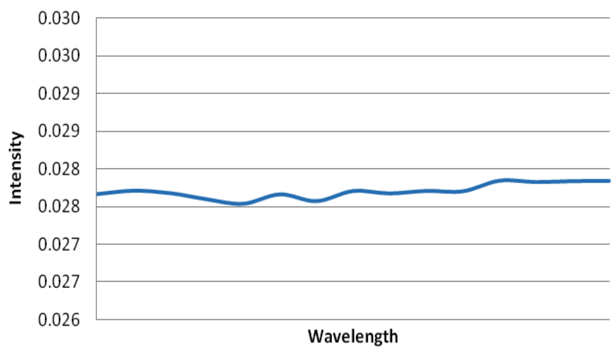
NIR technology in sorting equipment is often used for the separation of waste products to recover valuable materials by means of NIR spectroscopy detection. Most materials have a unique near infrared spectral signature and this can be used to distinguish different materials from each other, e.g. paper from plastics as well as within a material group such as PP vs. PET. The response spectrum of each single object is analysed and the material type is identified within milliseconds by the scanner and computer unit. Nevertheless, NIR detection has its limits, for example many black objects typically cannot be detected with this technology. The detection response of a variety of commercially available black plastic trays in the UK marketplace was assessed by NIR spectroscopy in a series of trials to check that NIR spectroscopy was not able to identify the plastic material and therefore the results would validate the assertion present in literature.

NIR assessments were performed at TITECH GmbH Germany to determine whether any black packaging from the samples could be recognised by a TITECH Autosort system, which utilises NIR spectroscopy, for polymer type. The detection rates were calculated using the TITECH standard classifier for polymers, without major optimisation for the specific material samples.

In total, eleven commercial samples were gathered from UK retail stores and tray manufacturers supplying the UK market. The samples included black coloured pots/tubs, tinted and sleeved bottles, tray packaging and also black multi-layer packs. Tray materials included, APET, CPET, rPET, HIPS, PP, and starch based polymer. These had been coloured with an unknown amount of carbon black pigment. Of the samples tested, none of the commercial black packaging products could be identified by polymer type. Figure 42shows an image and spectra from just one of these as an example.

Figure 42. Example of one of the black coloured tubs tested using NIR spectroscopy

Picture of black coloured PP tub on sort belt.



NIR spectrum showing low spectral intensity of the black tub

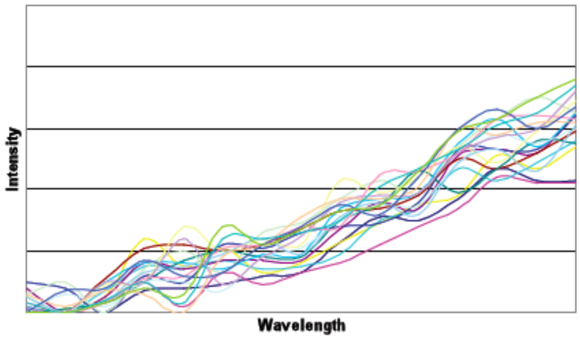
The NIR sensor could not identify this PP tub, as the signal intensity was not strong enough to give a clear spectral reading. This black tub therefore cannot be sorted by polymer type under industrial conditions using an NIR sorting system.

Mid Infrared (MIR) Spectroscopy Technology & Testing

The samples being used for the MIR tests consisted of several trays with an unknown amount of carbon black. A static laboratory MIR spectrometer performed measurement of the spectra and material recognition testing. Tests were performed, taking measurements across the mid infrared spectral region.

Details of the testing facility and the wavelength range in which the measurement were performed are confidential as the process is in commercial development. The MIR tests were performed to determine whether MIR spectroscopy is a suitable technique for identification and sorting of black plastic packaging that is coloured with carbon black.

Figure 43. Example of black tray tested by MIR spectroscopy



This tray was able to be detected with MIR spectroscopy and could be automatically sorted for reprocessing from the waste stream if MIR spectroscopy was used for detection.

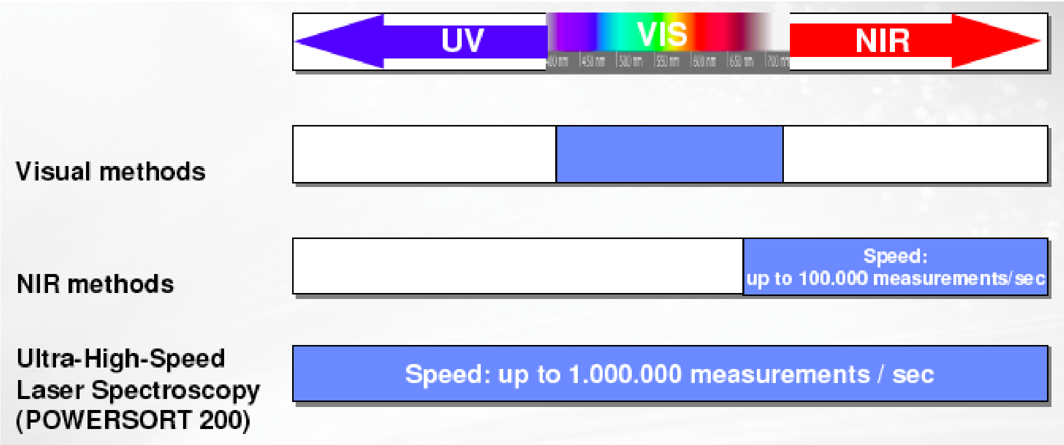
The MIR system showed a number of technical limitations that still need to be overcome. For example the tests showed spectra with low signal-to-noise of the black plastics due to localised melting of the black plastic trays when under the MIR light source, this issue needs to be further evaluated. MIR spectroscopy based systems also need further development in polymer identification speeds, so that the MIR systems can match those of current commercial NIR units and to achieve this sufficient cooling is needed to prevent melting of the polymer materials during the identification step.

The development of MIR automatic sorting equipment is still emerging and not yet available on a commercial scale in the waste-recycling sector however MIR spectroscopy may become an important sorting technique in the future.

Laser Spectroscopy Technology & Testing

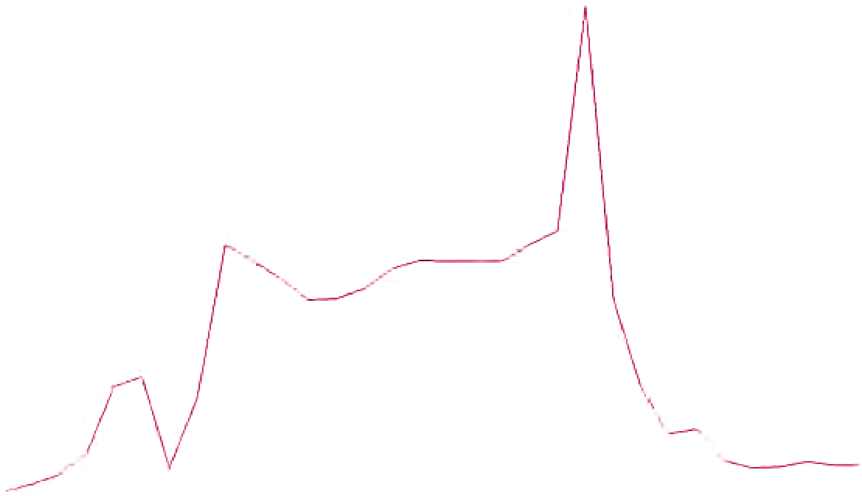
The Powersort 200 system from Unisensor utilises the material's overall emission spectrum, i.e. its physical "fingerprint", and is able to detect and separate it from the product flow. Up to 1,000,000 spectra are measured and evaluated every second. Due to its wide-band measurement ranging from the Ultra-violet (UV) to the NIR Powersort is able to detect and differentiate many different materials including some black materials.

Figure 44. Example of spectral range that the Unisensor Powersort 200 system measures to identify materials



This technique was selected for evaluation and testing as it is already being used for separation of different black plastics in the automotive area. According to Unisensor, it is able to achieve a high level of 99.7% purity after a sort and two re-sorting passes in an operation where 50% of the incoming flakes are black. If the percentage of incoming black flakes is significantly lower than re-sorting may not be necessary.

Figure 45. Example of spectra obtained for a black PET tray when analysed with Laser Raman fluorescence spectroscopy (Source: Unisensor GmbH)



The spectra obtained for the black PET tray sample showed a significant spectral response. Standard black PET trays can therefore be detected and differentiated with a laser spectroscopic system under industrial processing conditions and could potentially be recovered for reprocessing by MRF and PRF reprocessing facilities. While these systems are able to identify and sort plastics in flake formats, no systems currently operate commercially to sort whole items such as black pots tubs trays or bottles. Flake sorting systems are typically used in later (secondary) sorting stages at most plastics recycling operations, i.e. once the packaging items have been sorted by polymer type and colour.

Commercially, laser sorting systems are available from companies such as UNISENSOR GmbH and Visys NV. Unisensor use Laser Raman Fluorescence Spectroscopy and Visys NV systems utilise the reflection of laser light to perform detection. While economically viable in the high value PET recycling industry, they may not be economically viable for use purely for the purposes of recovering black plastic packaging.

Summary of Sorting Techniques for Black Plastics

Technical limitations make physical methods unlikely to provide a viable solution for commercial use. Markers were found to be too expensive and current systems are not yet suitable for high speed sorting systems. Plaques and trays containing standard carbon black colourant were tested by two commercial systems a NIR-based system supplied by TITECH, and a Raman (Laser) spectroscopic system supplied by Unisensor. These were also compared with results from a non-commercial (developmental) MIR spectroscopic system.

The commercial samples used for the tests were coloured with an unknown amount of carbon black. Under NIR spectroscopy the spectra were found to have very low intensity levels and appeared random i.e. without a unique spectral signature. The trial results proved that trays coloured with carbon black could not be detected by means of NIR-spectroscopy.

All the commercial trays tested showed a spectrum within the mid-infrared range and can therefore be detected using MIR spectroscopy technology. MIR spectroscopy therefore has excellent potential for identification and sorting of rigid plastic packaging coloured black with standard carbon black colourants. In summary, MIR spectroscopy was found to be a very promising identification technique but is still in commercial development and a number of technical and processing issues need to be resolved before the systems can become commercially available. The price of commercial MIR systems is currently not known but is expected to be higher than that of NIR systems and therefore MIR may not be economically viable for use purely for the purposes of recovering black plastics.

Using a laser spectroscopy system, the spectra obtained from the black PET, CPET and PS commercial samples were found to present significant spectra. These materials could therefore be readily detected and differentiated with this system under industrial processing conditions. Evidence from manufacturers suggests that laser techniques can achieve very high sort purity but trials proved that the systems could detect all commercial black trays but polypropylene and thermoplastic starch based trays showed lower spectral intensity. Unisensor have commented that since these tests have been performed the detection of black plastics including black polypropylene has been substantially improved and there is continued focus on improving the detection of black plastics.

The spectra obtained from commercial black PP and thermoplastic starch based trays showed low intensity and detection of the polymer type was difficult. Unisensor have reported that detection of black PP flakes at high speed is achievable but difficult, but there is potential for Laser Raman fluorescence spectroscopy system to accurately detect and sort larger articles such as black trays once commercial systems are developed.

The MIR system showed a number of technical limitations that still need to be overcome. For example the tests showed spectra with low signal-to-noise of the black plastics due to localised melting of the black plastic trays when under the MIR light source, this issue needs to be further evaluated. MIR spectroscopy based systems also need further development in polymer identification speeds, so that the MIR systems can match those of current commercial NIR units and to achieve this sufficient cooling is needed to prevent melting of the polymer materials during the identification step.

The laser spectroscopy based Unisensor Powersort 200 system returned positive identifications for PET and PS tray samples with standard carbon black colourant, but could not detect black PP and starch trays.

With NIR, TITECH found that, as expected, NIR could not detect any of the commercially available carbon black coloured trays. Moulded trial plaques compounded with varying levels of carbon black could not be detected even with addition levels as low as 0.1%, at which the samples already had a translucent black tint rather than an opaque black.

Conclusion

Based on these findings, and because most UK and EU plastic waste recycling facilities utilise NIR sorting equipment, NIR technology was selected as the basis for this project and was used to evaluate detection of alternative black colourants.

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Appendix 4: Food Contact Certificates of Compliance for Alternative Colourants

Colour Tone Masterbatch Ltd

Colour Tone Masterbatch Ltd

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www.colourtone-masterbatch.co.uk

<u>FOOD CONTACT DECLARATION</u>

Black 95493

We hereby certify that all ingredients of the above product are suitable for use in food contact applications under EU Directive 2002/72/EC (as amended) and Council of Europe Resolution 89)1.

K.L. Morgan

Kevin Morgan
QA Manager

Directors S: A Morris A Gaukroger
Pant Glas Farm Industrial Estate, Newport Road, Bedwas, Caerphilly CF83 8BJ UK

Reg Office: As above
Reg in England and Wales No: 3176153

ColorMatrix Group Inc – Food Contact EU Declaration of Compliance

ColorMatrix®

EU DECLARATION OF COMPLIANCE

Product Codes:	282-1011-5
Product Name:	Dye Black-5
Certificate Date:	24/03/2010 14:16:39

ColorMatrix Group Inc hereby declares that its product Dye Black-5 is a liquid masterbatch that may be lawfully used as a component of plastic articles that will contact food, subject to the restrictions provided below, in compliance with the legislation in place in the European Union (EU). Dye Black-5 meets the relevant requirements of European Union regulations 1935/2004 and 2023/2006 on materials and articles intended to come into contact with food.

Dye Black-5 is constituted solely of colorants and /or additives.

All colorant constituents of the ColorMatrix Group Inc product Dye Black-5 are compliant with the requirements and specifications of the Council of Europe (CoE) Resolution AP (89) 1 "On the use of colorants in plastic materials coming into contact with food".

All additives used in the ColorMatrix Group Inc product Dye Black-5 have a suitable listing under EU Directive 2002/72/EC Annex III (and amendments 2004/19/EC, 2005/79/EC, 2007/19/EC, 2008/39/EC and regulation EC 975/2009) 'List of additives which may be used in the manufacture of plastic materials and articles'.

Applicable Restrictions (if any)

Additive 1 is listed under European Directive 2002/72/EC with an SML of 0.20mg/kg

Only to be used in PET, polystyrene (PS), high impact polystyrene (HIPS) and polyamide (PA).

General Conditions

The present certificate does not warrant against modifications of Dye Black-5 resulting from its processing or from the addition of other products, nor against any inadequate use and/or storage of Dye Black-5 or the materials and articles containing it.

With respect to those constituents that are subject to restrictions, if any (see above), it is the responsibility of the finished article manufacturer to ensure that all restrictions, and organoleptic requirements, are met by the finished article and that such article is fully compliant with all the relevant EU and member state legislation.

When conducting migration testing, information on the composition of products may be required. ColorMatrix shall disclose such information when necessary, to the laboratory responsible for testing if a suitable confidentiality agreement is entered into.

This document is generated automatically therefore has no signature.

PRODUCT STATEMENT

Product Codes:	282-1011-5
Product Name:	Dye Black-5
Certificate Date:	24/03/2010 14:16:39

ColorMatrix Group Inc hereby declares that the product Dye Black-5 does not intentionally contain the following: Lead, Mercury, Cadmium, Hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers.

Furthermore, all raw materials used for the manufacture of Dye Black-5 are expected to meet the heavy metal requirements of Article 11 of European Directive 94/62/EC on packaging and packaging waste and the USA CONEG regulation. These require that the total of Lead, Mercury, Cadmium, Hexavalent chromium in packaging shall not exceed 100ppm.

General Conditions

The present certificate does not warrant against modifications of Dye Black-5 resulting from its processing or from the addition of other products, nor against any inadequate use and/or storage of Dye Black-5 or the materials and articles containing it.

It is the responsibility of the article manufacturer to ensure that all restrictions are met by the finished article and that such article is fully compliant with any applicable legislation. ColorMatrix Group Inc does not routinely test its products fo Heavy metal content and this does not form part of the specification.

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