

## Development Of A Standard Testing Protocol FOR Pet Recyclate and Methods Of Adding Value To The Post Consumer Resin

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**Abstract:** Packaging material has a very negative perception with consumers and environmentalists. It is perceived to be a waste of resources and a significant contributor to the growing levels of wastes. In addition it is often also linked to litter issues. Politicians are very aware of this with the result that pressure has been, and continues to be applied on packaging through the introduction of legislation in Europe, the USA, Japan and other countries around the world. In addition, recycling is seen by many as the most important recovery route and therefore the one that should take precedence (Recoup, 2009). The main aim of this industry co-funded project (Shabra) is to develop a suitable specification data sheet for Shabra so that the quality of its poly(ethylene terephthalate), [PET] recyclate can be ascertained. The Shabra Group is one of the few recycling companies reprocessing waste plastic materials into recycled products in Ireland. This research has included investigating the properties of recycled PET flake and determining the quality of the material (Ms. Fiona McCoole *et al.*, 2013).

**Key words:** Poly (ethylene terephthalate), Shabra Group, Specification data sheet

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### INTRODUCTION

Polyethylene terephthalate (PET) has become the most favourable packaging material world-wide for water and soft drinks bottles. The reason for this development is the excellent material properties of the PET material, especially its unbreakability and the very low weight of the bottles compared to glass bottles of the same filling volume. In comparison to other packaging polymers, PET has also a high clarity as well as good barrier properties towards moisture and oxygen. As a consequence, PET has mainly substituted glass as packaging material, but also metal cans. Nowadays, PET bottles are used for soft drinks, mineral water, energy drinks, and ice teas as well as for more sensitive beverages like beer, wine and juices. The amount of PET bottles for beverages is still increasing world-wide (Frank, W., 2011).

Poly(ethylene terephthalate) (PET) is the most recycled domestic packaging polymer. It is widely known that the majority of plastics waste continues to reach landfill, with 63.8% of plastic packaging waste still being sent to landfill in Ireland each year (Ms. Fiona McCoole *et al.*, 2013). At this rate landfill sites in Ireland will be at capacity within 3 years. The overall estimated amount of plastic waste generated following consumption and use of plastic materials was estimated to be 482,366 tonnes for the Island of Ireland. Landfill of plastics is still the most prevalent waste management option with 71% of plastic waste landfilled (Rx3 2011).

The development of a specification sheet included a review of PET specification sheets currently employed within the recycling industry and establishing test techniques most suitable in addressing Shabra's current industry needs. Properties including bulk density, alkalinity, moisture content and maximum contaminant levels were analysed. All the testing and analyses were carried out in accordance with recognised international standards including European Standard EN 15343:2007 (BS ISO 12418-2). Based on this research, techniques most suitable to Shabra in terms of reproducibility, equipment cost and ease-of-use were established. Following this phase of work, the quality of the PET recyclate was further determined.

### MATERIALS AND METHODS

#### **Materials:**

The materials and methods used in this research were selected in order to obtain data that can be correlated to real time industrial recycling processes. The samples used in this study were directly obtained from industry which assured results were relevant to industrial conditions.

A large amount of sample was required for the various tests performed in this study. Clear/blue PET (poly(ethylene terephthalate)) flake sample provided by Shabra Plastics was used for all the analytical tests. PET virgin samples were provided by Shabra Plastics and JL Goor Materials Ltd. PVC (poly(vinyl) chloride) sample in pellet form was supplied by Athlone Institute of Technology Materials Research Institute. All materials were used as received unless otherwise stated.

**PVC And Impurities Identification:**

Weighed exactly 1000g  $\pm$  0.1g of PET flakes and distribute them evenly in an aluminium (or metallic) container. Placed the container into an oven preheated to 220 °C and leave it at that temperature for one hour. After the necessary time for the cooling of the sample, inspect it accurately. It was then inspected for contaminants using the colour guide of:

- Black carbonised particle = PVC
- Yellow particle = Polyolefin
- Brown/burnt = cellulose and paper

The impurities content of sample was calculated as ppm (parts per million) for each impurity.

**Moisture Test:**

A 50g of sample (Mo) was weighed out in to a porcelain crucible previously dried and calibrated to constant mass. The crucible was placed in an oven @ 150°C for 4 hours. The crucible was cooled in a desiccator for 30 minutes and weighed (M1). The difference between the initial and the final mass of the test portion is used to calculate % moisture.

To calculate % moisture in samples the following formula was used.

$$\%H_2O = [(M_o - M_1) \div M_o] * 100. \quad \text{Equation 1}$$

**Bulk Density:**

PET flakes filled in a known volume container up to the brim are weighed. The bulk density is derived from the container volume and the mass of the PET recyclates. The volume of the container was calculated by filling the container with water and by regarding 1g as 1cm<sup>3</sup>. The mass of container (W1) was weighed to the nearest 0.1g. The container was filled with sample to the edge of container (overflowing) and after tapping the edge of container by fingers thrice, levelled the sample along the edge of container using a ruler and weighed the mass of container (W2) to the nearest 0.1g.

The bulk density was calculated according to the following formula.

$$Bd = [(W_2 - W_1) / V] \times 10^3 \quad \text{Equation 2}$$

Where

Bd = bulk density (kg/m<sup>3</sup>)

W1 = mass of container (g)

W2 = mass of container and sample (g)

V = volume of container (cm<sup>3</sup>)

**Residual alkalinity (pH testing):**

A given quantity of sample was mixed by stirring with a measured volume of distilled water. The residual soda present on the surface of the PET recyclates produces an increase of the pH of the water, which can be measured through a combined glass electrode.

The pH meter calibration was carried out using buffered solutions with a known pH. Exactly 100g of the sample was weighed in to a 1000mls beaker and 500mls of distilled water was added, whose pH was previously determined. The pH should be between 6 and 8. The solution was stirred for 10 minutes ensuring all the PET recyclates are completely immersed in the water. After the stirring was stopped and the solution decanted, the test electrode was immersed and the pH, when stabilized was recorded. The result of the analysis was reported as the pH of the solution, by comparison with the initial pH of the distilled water.

## RESULTS AND DISCUSSION

Loads of PET are being produced and used in the world every day. While PET recycling has steadily increased over the years, the percentage of recycled material used for food grade applications has been low compared to other applications. The focus of this research is to understand the implications with respect to the PET recycling process. PET is a very valuable raw material (Market price of up to \$1/lb. in 2011) and finding newer applications such as replacing traditional raw materials, metals and resins. rPET has a positive impact on the environment as it reduces the impact on landfills, consumer waste, reduces emissions and helps in energy conservation (Rajakutty, A., 2010).

**PET Recycling Processes:**

There are two major processes used for recycling PET, (1) chemical and (2) mechanical recycling. The chemical recycling process involves depolymerisation reactions into monomers or partial depolymerisation into oligomers. The compounds arising from depolymerisation reaction include terephthalic acid, dimethylterephthalate and bis-(hydroxyethyl) terephthalate. Mechanical recycling is the most widely used

method because it is easier, faster and much more economical compared to chemical recycling. The mechanical recycling process has several stages such as bottle collection, bailing unpacking/sorting, grinding, flake washing, drying of material and removal of labels.

As Shabra Plastics use the mechanical process to recycle plastic polymers the research focused on this process and its implications with respect to PET recycling. The techniques most suitable to Shabra in terms of reproducibility, equipment cost and ease-of-use was established. Properties including bulk density, alkalinity, moisture content and maximum contaminant levels were analysed. All the testing and analyses were carried out in accordance with recognised standards including European Standard EN 15343:2007 (BS ISO 12418-2). To date this has yielded valuable information relating to the efficiency of each step in the recycling process and resulted in cost savings and improved quality recycle.

#### **PVC And Impurities Identification:**

Contamination of POSTC-PET (post consumer PET) is the major cause of deterioration of its physical and chemical properties during re-processing. It is a requirement to minimise the amount of contaminants present as it leads to better R-PET (recycled PET) quality. The most harmful acids for the recycling process are acetic acid, which is produced by poly(vinylacetate) degradation, rosin acid and abietic acid that are produced by adhesives and hydrochloric acid that is produced by PVC. The acids act as catalysts for the chain scission reactions during POSTC-PET melt processing. It has been reported that the presence of PVC increases PET chain scission due to the catalytic effect of hydrogen chloride evolving during the degradation of PVC. The presence of PVC also results in discoloration of PET during processing (Rajakutty, A., 2010).

As part of this study there was a requirement to be able to quantify and identify the contaminants present. By being able to record the quantity of contaminants per sample allowed the company to be able to determine the quality PET recycle for customer. For the determination of contaminants present during the study, see Table 1 for specifications required for different applications (www.CWC.Org).

**Table 1:** Summary of determination of contaminants

Specification	PVC (ppm)	Labels (ppm)	Colour PET (ppm)
Fibre	200	10	5000
Bottle	10 - 25	10	100 - 1000
Strapping	0 - 20	10	0 - 10

The results obtained from both industry and AIT were similar which demonstrated that the method is robust to be used in industry to quantify contaminants present in flake samples.

#### **Moisture Test:**

Water reduces molecular weight during PET recycling through a hydrolysis reaction. Most water content comes from the flake washing process but can be reduced substantially by proper drying. Drying is regarded as an essential step in PET recycling. Minimising the moisture content of PET flakes reduces the hydrolytic degradation effect and leads to higher R-PET melt strength (Awaja and Pavel 2005). The drying process also gets complicated since the drying speed decreases with increasing thickness of the flakes and increasing level of crystallisation (Thiele, D.U. & D.S. Gneuss, 2004). See Table 2 for a summary of moisture specifications.

**Table 2:** Summary of moisture specifications

Application	Fibre	Bottle	Strapping
% Moisture	< 1.0	< 0.6	< 1.0

#### **Bulk Density:**

Bulk density is the weight per unit volume of the raw plastic material as it is purchased from the material supplier. Bulk density is useful as an incoming quality check on material. Careful measurement can reveal lot to lot variations and a significant change in bulk density would indicate that there is variation in weight and/or size.

PET flake was tested numerous times for bulk density analysis and an average value meeting fibre specification was obtained.

**Table 3:** Bulk density guideline specifications

Food Grade	Non-food Grade	Fibre Grade	Bottle Grade
300-500 kg/m <sup>3</sup>	250-500 kg/m <sup>3</sup>	300-400 kg/m <sup>3</sup>	288-481 kg/m <sup>3</sup>

#### **Residual alkalinity (pH testing):**

During the mechanical recycling process a large number of contaminants are present on the flakes which need to be removed. The flakes are washed with hot dilute alkaline water. This helps to remove the volatile and non-volatile contaminants on the surface since the surface gets hydrolysed. Alternative methods to wash the flakes include washing with aqueous hot washing with 2 % Sodium Hydroxide (NaOH) solution and a detergent

and then following up with cold wash (Rajakutty, A., 2010). It is necessary to ensure that the flakes are well washed after treatment and by using the residual alkalinity test ensures that the wash process is sufficient.

**Conclusion:**

This research has provided a structure for the Shabra group to determine the quality of their PET recycle flake and also a capacity to determine the efficiency of the several steps involved. The quality of the raw material for recycling is one of the biggest factors for the high contaminant levels present in the PET flake after the mechanical recycling process. The recycling process involves several steps which the PET flakes are exposed to different temperatures and mechanical processes which caused contamination, degradation and loss of properties (Frank, W., 2011; Rajakutty, A., 2010). Efforts are on-going to further improve the quality of the recycle so that the material may be sold to higher value end markets and reduce material reaching landfill.

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