

# **Solvent extraction as additional purification method for post- consumer plastic packaging waste**

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Report 1261

## Colophon

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## Abstract

An existing solvent extraction process currently used to convert lightly polluted post-industrial packaging waste into high quality re-granulates was tested under laboratory conditions with highly polluted post-consumer packaging waste originating from municipal solid refuse waste. The objective was to study the technical feasibility of using this extraction technology and to study the quality of the produced cleaned plastic flakes. Two types of dirty plastic flakes from household waste were used; transparent to white flexible film flakes and PET-bottle flakes. The flexible material was recovered from household waste with a wind-sifter and a Filmgrabber. The flexible plastic packaging waste was subsequently hand-sorted on colour, shredded and tested. The PET-bottle material was recovered from drum-sieved MSRW from which rigid packages were recovered with a NIR (PE, PP, PET positive) sorter and which were subsequently NIR sorted into a PET-product, which was subsequently hand-sorted into transparent to light blue PET bottles that were shredded.

The ethyl acetate based extraction process made the flexible material optically clean; white flakes were recovered with hardly any printing left, no foreign materials detectable and no smell (even no garbage smell). The process removed dirt, soil, prints, lacquer, paper fibres, glue, etc. from the surface.

The PET-bottle flakes were recovered as shining flakes, without smell. The process had removed glue residues, dirt, soil, etc. from the surface and had clearly improved the optical appearance and smell.

This result implies that the studied extraction technology can play an important role in a future total process for the re-processing of plastic packaging waste that has been recovered from municipal solid refuse waste into high quality re-granulates. Nevertheless, a matching overall process will have to be engineered and the extraction technology can also be optimised within the context of such a new overall process. Furthermore, such a new overall process with this extraction technology, is currently not available at a commercial plastic waste re-processing plant and will, therefore, still have to be established.

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# 1 Introduction

The objective of this study is to evaluate the technical feasibility of a solvent extraction process with laboratory means for the cleaning of milled goods made from plastic packaging waste which has been recovered from municipal solid refuse waste (MSRW).

This extraction technology has run since 15 years in an industrial packaging plant, near Osnabrück, in the North of Germany. In this plant industrial PE film waste is being shredded and treated with this extraction technology. The produced plastic film flakes are being reused in 50 kg chemical bags, 30, 45 and 70 litres compost bags and shrink films for securing transport loads. This process is known as the Nordenia Extraction and Cleaning process or NorEC. [Brandrup 1996] One of the original developers of this technology was present during our trials to reproduce his extraction technology in a laboratory scale with laboratory means. Additionally he explained the modalities of the technology, some of which are listed below:

- In order to obtain high quality re-usable plastic materials after this treatment technology the input materials need to be well sorted in type of plastic and base colour. Impurities like dissolvable plastics (PVC, PS, CA, etc.), sticky tape, physically trapped particles, etc. can affect the quality of the product negatively. A previous sorting step in base colours renders a cleaned product after the process with a higher value and potential higher re-use potential.
- The commercial process is run in a carrousel extraction unit in which there are 17 chambers with a cross-flow set-up, meaning that the hot ethyl acetate solvent is first used to clean in the most clean compartment and then flows on in the opposite direction of the plastic material. In this extraction device the plastic particles are agitated vigorously in the hot ethyl acetate solvent. Furthermore, the ethyl acetate is run over the plastic particles in such a way that impurities can be dissolved from the surface of the plastic particles, but that the penetration of the dissolved material into plastic material is limited.
- The use of hot ethyl acetate in relative large quantities implies that the extraction is performed in a well-ventilated room to minimise fire risks and health risks for the employees and that the solvent is constantly regenerated by distillation and reused.
- This treatment will typically remove the following materials from the plastic particle surface: dust, dirt, anti-blocking agents, nitrocellulose lacquer and contained pigments, glue residue, degraded plastic components, various plastic additives, etc. This treatment does not remove UV-radiation cross-linked prints.

The related company IPP BV (International Pure Propulsion) in Stadskanaal has the opinion that this extraction technology is well suited to clean plastic particles that have originated from MSRW recovery. In the opinion of this company plastic packaging waste that has been separated off from MSRW should be treated by a complete process of which this extraction technology should be an important process step. [Von Deym 2000] Overall, the plastic material should be

separated in main plastic type by near-infrared sorting (NIR), then being sorted in a few base colours and being sorted in main packaging types. Subsequently the plastic material should be shredded, washed with hot water, dried, extracted with ethyl acetate, dried and reformulated into re-granulates. Most of the steps in this whole process are technically common and feasible. The step of automatic sorting in packaging types is not straight-forward and –in our perspective– perhaps not always necessary, but we are also confident that with ballistic separators and wind sifters some separation in basic packaging styles can be made. This leaves the solvent extraction technology in our opinion as technically the most innovative and challenging. Therefore, it was decided between IPP and Food & Biobased Research (FBR) to perform a test with some unwashed plastic milled goods originating from household waste with this extraction technology in a laboratory setting. FBR would perform the extraction and report on the results and IPP together with the original inventor would advise us on how to mimic the commercial process as close as possible under laboratory conditions. The extraction tests were conducted on May 25<sup>th</sup> 2011 in Wageningen. This report describes the extraction tests performed and products obtained.

## 2 Methods

### 2.1 Materials

Ethyl acetate was used as extraction solvent. 25 litre of technical quality was purchased from Boom BV in Meppel the Netherlands.

The plastic film material originated from municipal solid residual waste. It was recovered from MSRW in Groningen by the Attero recovery facility in 2010. It has been stored at 7°C in Wageningen. It is composed of about 20% paper and board and about 73% polyethylene based film material and 7% polypropylene based film material. This material was manually separated into a fraction of transparent, printed-transparent and printed-white flexible plastic materials. A sample of roughly 10 kg of this sorted flexible material was shredded at the RWTH in Aachen with a rotary shear shredder to film flakes of 1-4 cm dimensions. This material was visually dirty and also smelled, see Figure 1.

The PET-bottle flakes originated from Dutch and Spanish municipal solid residual waste. It was recovered by two facilities; Attero in Groningen and Ecoparc in Barcelona. The rigid plastic fraction was NIR sorted in Wageningen in a PET, PE, PP, PS and PVC fractions. From the PET fractions the transparent to light blue PET bottles were hand sorted. These transparent and light-blue PET bottles were shredded at the RWTH in Aachen with a rotary shear shredder to flakes of 2-6 cm dimensions. This material was unwashed and hence dirty, see Figure 1.



Figure 1: Transparent shredded film material (left) and Shredded transparent-to light blue PET bottle material (right)

### 2.2 Experimental set-up

The set-up was composed of a 6 litre round-bottom three neck flask which was equipped with a mechanical stirrer through the centre neck and a reflux cooler on a side neck. The third neck was blocked with a stop and used to fill and empty the flask. A picture of the set-up is shown in Figure 2. The flask was heated with an isomantle electrical heater, which was positioned on a

removable lab.-table. This set-up allowed the contents to boil under reflux-cooling and to empty the contents over a Büchner-funnel into a vacuum Erlenmeyer.

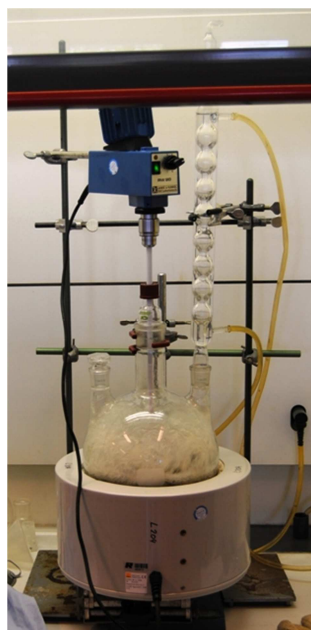


Figure 2: The applied set-up.

After boiling the extract was filtrated with Wattmann 100 paper filters. The residue was collected and dried in an aluminium cup, the filtrate was distilled in film evaporator. The distilled ethyl acetate was re-used. The filtrate was concentrated into an aluminium cup and dried.

The last step in the process was a conventional steam-distillation to remove solvent. The flask with extracted plastic flakes was filled with water and heated to boil. The reflux cooler was removed and replaced for a distillation set-up, a glass cooling pipe and a collection Erlenmeyer, see Figure 3.

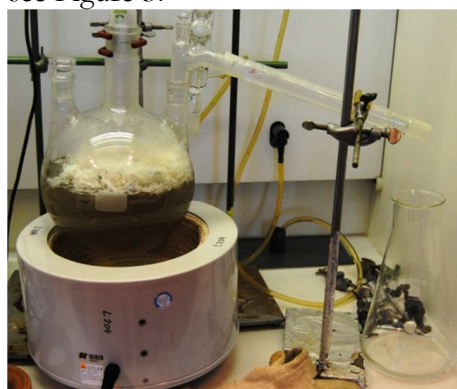


Figure 3: Set-up just after steam-distillation with a distillation set-up, cooling tube and collection Erlenmeyer.



### 2.3 Extraction protocol

The flask was filled 50 to 100 gram of plastic flakes and 2 litre of ethyl acetate. The contents was heated and stirred as fast as was possible. The contents was vigorously stirred and boiled for 15 minutes, after which the heating mantle was removed and the hot solvent was poured over the Büchner funnel. The filtrate was placed aside to allow for cooling and settling. Most of the plastic remained in the flasks. The few bits of plastic on the Büchner funnel were re-inserted into the flask. Subsequently, 2 litre of pre-heated ethyl acetate was introduced into the flask and the stirring and heating recommenced for another 15 minutes. This was repeated to have in total maximally five extraction cycles with ethyl acetate.

Finally, 2 litre of pre-heated water was introduced into the flask and the reflux cooler was changed for a distillation set-up, glass cooling pipe and a collection Erlenmeyer. The mixture was heated and vigorously stirred and allowed to distil for about 15 minutes. The smell of the distillate was monitored. The initial smell was strong “ethyl acetate” and after about 15 minutes this solvent smell was gone and the steam distillation was stopped. The contents of the flask were emptied over a Büchner funnel. The aqueous filtrate was left to cool. The plastic particles were first dried in a normal oven at 75°C to remove the water and secondly in a vacuum oven at 40°C and 50 mbar overnight. Finally the weight of the dried and recovered plastic was measured. The ethyl acetate extracts were all filtrated. The residues were collected in aluminium cups, dried in an oven and weighted. The filtrates were distilled using a film evaporator and the residual concentrated mass was transferred into aluminium cups, dried in an oven and weighted. The aqueous filtrate of the steam distillation was only filtered and the residue was collected in an aluminium cup, dried in an oven and weighted.

### 3 Results

#### 3.1 Transparent to white plastic packaging film waste

The dirty plastic packaging film waste (51.27 gram) was extracted four times with 2 litres of hot ethyl acetate. After the extractions and oven drying 44.68 gram of purified plastic film waste was recovered. Hence the yield was 87%.

The quality of film waste is enormously improved in terms of colour and odour. The colour of the film material was changed from a dirty brown-grey for the untreated material to an almost white to transparent material. With the exception of some fully coloured film pieces all printed-on colours appear to have been removed. Additionally, the typical municipal waste odour of the starting material is absent on the purified material.



Figure 4: Photo's to compare untreated and treated film material (left) and a photo of one of the last process steps the filtration after steam distillation (right).



Figure 5: The ethyl acetate extracts of the film material (back row from left to right 1 to 4 + steam distillation water) and of the PET bottle material (front row from left to right 1 to 3 and steam distillation water)

A picture of the collected ethyl acetate extracts is given in Figure 5. The dried residues and filtrates of the extraction of plastic film material are shown in Figure 6. The weights and descriptions of these removed materials are summarised in Table 1.

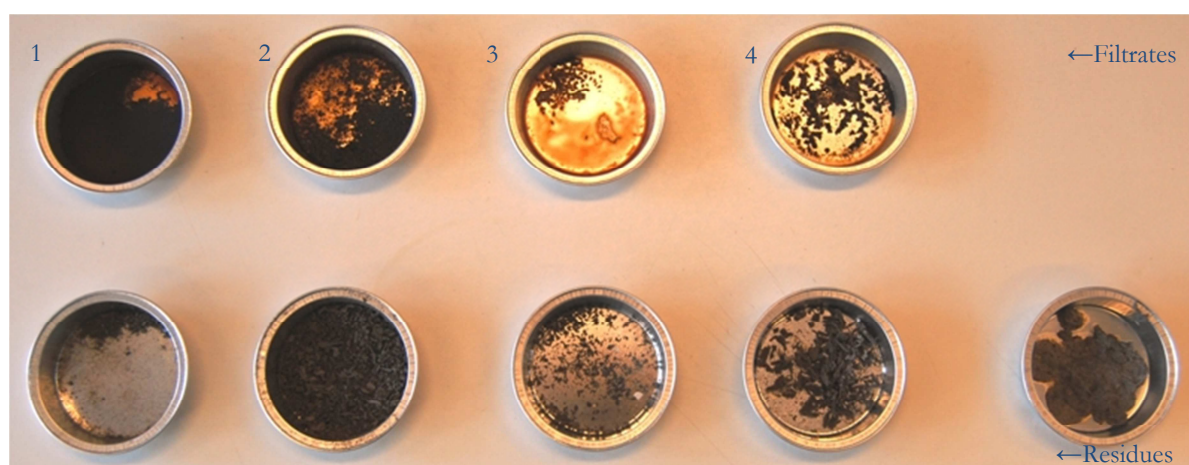


Figure 6: The concentrated filtrates (above) and the residues (below) from the film flakes. Left is the material from the first extraction cycle and to the far right is the residue of the last steam extraction.

The filtrates were concentrated and dried. In total roughly 1 gram of concentrated filtrate was collected. This is a dark-red waxy material is likely to be composed of nitrocellulose lacquer, degraded polymer fragments, possibly polymer additives and various waste components. Also about 2 gram of residue was collected which is a grey material composed of mineral pollutants and paper fibres. Since 3 gram of materials were removed from 44.68 gram of cleaned plastic film pieces the initial pollution level would have been roughly 6%. In case the moisture loss of the input material is also considered within the loss, it adds up to 13%.

Table 1: Weights and descriptions of the filtrates and residues of the extracts originating from dirty plastic film material.

Sample	Weight, [gram]	Description
PE-1 Filtrate	0.2551	Dark red waxy material
PE-1 Residue	0.2176	Grey paper fibres and ash
PE-2 Filtrate	0.4902	Dark red waxy material
PE-2 Residue	0.7122	Grey paper fibres and ash
PE-3 Filtrate	0.1399	Dark red waxy material
PE-3 Residue	0.0906	Grey paper fibres and ash
PE-4 Filtrate	0.1080	Dark red waxy material
PE-4 Residue	0.3514	Grey paper fibres and ash
PE-ST Residue	0.7035	Fluffy paper fibres with some grey ash
Total	3.0685	

### 3.2 Transparent to light blue PET bottle waste

The dirty transparent to light blue PET bottle waste (77.59 gram) was extracted three times with 2 litres of hot ethyl acetate. Since the extracts were only lightly coloured, three times was considered enough. After the extractions and oven drying 76.9 gram of purified PET bottle waste was recovered. Hence the yield was 99%.

The quality of PET bottle waste is clearly improved in terms of colour and odour. The original PET bottle material contained two types of visible impurities; normal dirt (soil, dust, etc.) and thin layer of a fatty greyish material. Both types of impurities are removed with this extraction process. The cleaned PET has clearly less dark colours and it shines; reflecting the light much stronger. Furthermore, the initial PET bottle waste material did carry a limited but present waste odour. This odour was absent after the cleaning process. After the vacuum oven treatment even the ethyl acetate odour was absent and no clear odour could be detected.



Figure 7: Photos of the PET bottle material. The cleaned PET material is shown on the left and a comparative photo between the non-treated and treated PET is shown on the right.



Figure 8: The concentrated filtrates (above) and the residues (below) of the PET bottle flakes. Left is the material from the first extraction cycle and to the far right is the residue of the steam extraction

The ethyl acetate extracts were filtrated, concentrated and dried. The photo of dried filtrates and the residues are shown in Figure 8. The weights and descriptions of these dried waste products that were removed from the PET bottle material are given in Table 2.

**Table 2: Weights and descriptions of the filtrates and residues of the extracts originating from dirty PET bottle waste material.**

Sample	Weight, [gram]	Description
PET-1 Filtrate	0.2551	Red waxy material
PET-1 Residue	0.2176	Grey fibres, some sand grains
PET-2 Filtrate	0.1038	Orange waxy material
PET-2 Residue	0.0493	Grey fibres
PET-3 Filtrate	0.0355	Yellow waxy material
PET-3 Residue	0.0114	Grey fibres
PET-ST Residue	0.0625	Grey paper fibres
Total	0.7352	

About an equal mass of fibrous material and waxy material were removed from the waste PET flakes. Most of this material is likely to origin from the waste it has been in close contact with during the collection. Only the glue used to connect the labels to the bottles is also likely to be present in the extracted waste material. In general, this treatment clearly improves the outer appearance of the waste PET flakes and the smell.

## 4 Discussion

This research has clearly demonstrated that dirty plastic particles originating from municipal solid refuse waste can be cleaned thoroughly to produce optically clean plastic particles. These cleaned particles have no detectable smell. This implies that the extraction process has removed the odorous components that give rise to the typical light-acid garbage odour which was present on the input material. The input materials used were worst-case examples; originating from municipal solid waste with only a manual sorting step to remove other waste, other plastics and other packaging objects with different colours. Hence, they were not washed with hot water to remove adherent dirt and soil, meaning that the input materials can be considered as worst-case dirty plastic packaging waste particles. Even with this dirty input material, the laboratory process applied was able to remove both the dirt and soil and also fatty, waxy pollutants, some type of printing, degraded plastic material and odours.

This report has shown that the extraction technology is technical feasible. It does not report on the economic feasibility of this process. Nevertheless a German company has 15 years of operational experience with this technology and runs it on a pure commercial basis with post-industrial LDPE-waste. The costs of their extraction process has been documented in 1996 to be 300 DM/ton. [Brandrup 1996]

It should be stressed that this extraction technology is a part of the overall process and not a panacea for everything. It shows that dirty plastic packaging waste flakes from MSRW can be cleaned to highly purified plastic flakes (without optical and odorous impurities). The final result of a future process will, however, not only depend on this one step, but from a chain of steps that form the overall process and all these steps will need to be carefully engineered. This future overall process is currently not available at a plastic packaging waste re-processing plant and should still be established.

This extraction technology is relatively sophisticated and demanding in comparison to most of the currently applied technologies in the plastic waste re-processing industry. This means that many incumbents will either not be able to include such a technology in their process, or that they will have to upgrade their facilities, environmental permits, fire-prevention policies, educate employees, etc.

The high quality plastic flakes obtained from this process in terms of optical qualities and odour does not automatically mean that this quality would also be sufficient for food grade applications. The research conducted was not intended to be a part of an evaluation of a total recycling production process to qualify for EU directive 2008/282 for food-contact applications. Although there is no doubt that a total recycling process which includes this solvent extraction technology might qualify



## 5 Conclusions

Dirty milled plastic goods originating from municipal solid refuse waste that have been sorted on plastic type (NIR), colour and subsequently have been shredded to flakes can be cleaned thoroughly by an extraction process with hot ethyl acetate solvent. This extraction process removes various impurities from the surface of the plastic flakes, including fatty substances, some types of printing ink, degraded polymer residues, soil and dirt. After the treatment the plastic flakes are optically clean, shining (full reflective) and odourless. This extraction process was executed on a laboratory scale (to mimic the industrial process) with plastic waste samples originating from real municipal solid refuse waste.

This extraction technology can play an important role in a future total process for the re-processing of plastic packaging waste that has been recovered from municipal solid refuse waste into high quality re-granulates. The matching overall process will have to be engineered and the extraction technology will have to be optimised within the context of such a new overall process. Such a new overall process, is currently not available at a commercial plastic waste re-processing plant and will, therefore, still have to be established.

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