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Study on the Feasibility of Hazardous Waste Recycling: The Case of Pharmaceutical Packaging

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1. Introduction

Hazardous waste management should fulfil the following three main goals: (i) to protect human health and the environment, (ii) to reduce waste while conserving energy and natural resources and (iii) to reduce or eliminate the volume of waste to dispose of. The last two of these goals may derive from recycling, which aims at reducing raw materials and energy consumption and decreasing the volume of waste materials that must be treated and disposed of.

However, recycling must be conducted in a safe way, ensuring human health and environment protection. Recycling activities should be regulated at a different degree on the basis of the risk they cause to human health and the environment. A hazardous waste destined for recycling must be identified by type and recycling process in order to determine its level of regulation (Linninger & Chakraborty, 2001).

Pharmaceutical packaging represents a very small percentage of hazardous waste, but its management can cause problems for the environment, depending on the type of packaging waste is concerned (Sacha et al., 2010). Such waste may include:

- uncontaminated waste (assimilated to domestic waste: paper, cardboard, glass, plastic);
- contaminated waste (paper, cardboard, glass, plastic), e.g. waste that has been in contact with cytotoxic products, blood, blood-derived products or radioactive products.

Waste is created at all stages of the supply-chain: production, distribution and use of a pharmaceutical product. At each step, care therefore needs to be taken, either by the manufacturer or the end-user, to protect the environment (Biniecka et al., 2005; Dillon & Rubinstein, 2005).

In several European countries, pharmaceutical manufacturers must dispose of their waste, or by themselves or by external specialized companies, and are encouraged to recover packaging waste. In both cases, waste management represents a considerable cost for the manufacturers.
The use of environmental-friendly packaging (i.e. recyclable or degradable packaging) has to be considered. Valuable packaging materials, such as aluminium paper, glass and plastic materials, can be extensively recycled if they have not been in contact with toxic or dangerous substances (Bauer, E.J., 2009).

This chapter is focused on a feasibility study for the management of packaging waste from a pharmaceutical plant, considering the following phases:

- waste materials characterization;
- preliminary tests on waste processing;
- set up of size reduction (comminution) operations.

Experimental tests have been executed on several typologies of packing, as listed:

- primary packaging:
  - bottles in high density polyethylene (HDPE), for suspension to be reconstituted;
  - bottles in poly(ethylene terephthalate) (PET), for syrup;
  - plastic bags and films of varying composition and thickness;
- pharmaceutical waste:
  - flexible multi-layered (plastic and aluminium) sachets containing granular medicine.

2. Experimental

In a first stage, the products under investigation were characterized as derived from the industrial process. The results of the characterization were utilized to set up preliminary tests on waste processing, in particular the comminution operations were evaluated. Finally, an experimental plan was carried out to assess the feasibility of waste recycling.

2.1 Methods for characterization

The following methods were adopted in the experimental set-up for the characterization of products under investigation:

- image analysis, to measure geometric and morphologic characteristics; the results were evaluated by statistical methods;
- dry sieve analysis, to classify the size distribution of particles;
- laser granulometry, to classify size distribution of particles in the interval between 0.1 and 1,000 μm;
- infrared spectroscopy, to recognize chemical composition of polymeric materials under investigation.

2.1.1 Image analysis

The images for characterization were acquired by the stereoscopic microscopy Leica Wild M8 and a by a digital camera Olympus C-5060 Wide Zoom. The image analysis was carried out by the software SigmaScan Pro© Version 5.0.0 (Systat Software Inc., 2007), which provides a complete set of tools to analyse structure and dimension of an object’s image.

Firstly, calibration allowed to convert image unit from pixel to millimetre (Figure 1). After calibration, the image quality for data elaboration has been enhanced, increasing the
distinction between particles and background, by varying contrast, brightness and colour of the image (Figure 2). In the measurement process, the software automatically recognizes objects on the image (Figure 3) and computes geometric and morphologic parameters, accordingly to operator’s choice.

![Fig. 1. Calibration process to convert image unit from pixel to millimetre.](image1.png)

![Fig. 2. Variation of contrast, brightness and colour of an image.](image2.png)

![Fig. 3. Automatic recognition of objects on the image.](image3.png)
The following geometric and morphologic measurements were considered.

**Area:** reports the area in mm² for the selected object.

**Compactness:** reports a numeric non-dimensional measurement of the shape of an object. It is defined as the perimeter squared, divided by the area:

\[
\text{Compactness} = \frac{\text{perimeter}^2}{\text{area}}
\]  

(1)

The minimum Compactness of a perfectly measured and digitized circle is \(4\pi\) (about 12.57). As an object tends toward the shape of a line, the Compactness tends towards infinity.

**Major Axis Length:** calculates major axis of the object (defined by the two most distant points on the object) and reports the length in mm of the axis.

**Minor Axis Length:** calculates minor axis of the object (defined by the two most distant points on the object that creates a line perpendicular to the major axis) and reports the length in mm of the axis.

**Perimeter:** returns the perimeter in mm of an object.

**Shape Factor:** measures the shape (circularity) of a measured object. This non-dimensional measure is defined as \(4\pi\) times the object's area divided by the perimeter squared:

\[
\text{Shape factor} = \frac{4\pi \cdot \text{area}}{\text{perimeter}^2}
\]  

(2)

A perfect circle will have a Shape Factor of 1. A line's Shape Factor will approach zero.

**Feret Diameter:** describes the shape of an object. It gives the diameter of the equivalent circular object that has the same area as the current object. For each object, it calculates the theoretical diameter of the object if it were circular in shape. This measure is often compared with an object's major and minor axes lengths to create new shape parameters.

The results obtained from image analysis were evaluated by considering statistical parameters, as described in the following.

**Number of objects:** counts the numeric values in the considered set.

**Mean:** returns, as central tendency, the order of magnitude of the value for each considered measurement. The arithmetic mean \(\bar{x}\) is calculated as the sum of all measurements divided by the number of observations in the data set:

\[
\bar{x} = \frac{1}{N} \sum_{i=1}^{N} x_i
\]  

(3)

where \(x_i\) is the single measurement and \(N\) is the total number of measurements.

**Minimum:** returns the least value of the considered data set.

**Maximum:** returns the greatest value of the considered data set.
Standard deviation: shows how much variation or dispersion there is from the mean in the data set and it is measured in the same unit of the data. The standard deviation \( \sigma \) is directly derived from the variance \( \sigma^2 \), which unit is the square unit of the considered data, as its square root:

\[
\sigma_x = \sqrt{\frac{\sum_{i=1}^{N} (x_i - \bar{x})^2}{N}}
\]

where \( \bar{x} \) is the arithmetic mean.

Standard error: returns an estimation of the standard deviation \( \sigma_x \) of the estimator, giving a valuation of its imprecision. If the estimator is the arithmetic mean of \( N \) independent measurements with the same statistical distribution, the standard error is given via the equation:

\[
se = \frac{\sigma}{\sqrt{N}}.
\]

Confidence interval: refers to the range of values preceding or following a mean value where it is expected an unknown population parameter (e.g. the true mean) is located. The width of the confidence interval gives an indication about uncertainty of the unknown parameter. If independent samples are taken repeatedly from the same population, and a confidence interval calculated for each sample, then a certain percentage (confidence level) of the intervals will include the unknown parameter. The confidence level is the probability value \((1 - \alpha)\) associated with a confidence interval. For example, say \( \alpha = 0.05 \), then the confidence level is equal to 0.95, i.e. a 95% confidence level.

Let’s be the true mean the considered unknown parameter; the confidence level is given by:

\[
\bar{x} \pm A_{\text{conf}} \left( \frac{\sigma_x}{\sqrt{N}} \right)
\]

where \( A_{\text{conf}} \) is area under the normal distribution curve that is equal to the chosen confidence level. In the case under investigation confidence levels of 95% and 99% have considered.

2.1.2 Dry sieve analysis

The dry sieve analysis is a mechanical method to assess the particle size distribution. A set of sieves with wire mesh cloth is stacked in column, so that each lower sieve has smaller openings than the one above; at the base of the column there is a round pan. A representative weighed sample is poured into the top sieve. The column is typically placed in a mechanical shaker, that shakes the column for a fixed amount of time. After the shaking is complete the material on each sieve is weighed and divided by the total weight to gain the percentage retained on each sieve. In this study, certified high-precision sieves Giuliani in stainless steel (ASTM series) were utilized.
2.1.3 Laser granulometry

The laser granulometry analyses of the effect of diffracted light produced by a laser beam passing through a dispersion of particles. The angle of diffraction increases as particle size decreases. After mixing in distilled water (or alcohol), the representative sample is introduced in the measuring cell. The laser beam (wavelength = 632.8 nm; power = 5 mW) passes through the suspension and is deviated by particles accordingly to their particle size. The deviation is then analyzed by detectors. This method can measure particle sizes between 0.1 and 1,000 μm.

The laser granulometer utilized in this investigation was SYMPATEC HELOS/KA.

2.1.4 FTIR spectroscopy

The Fourier transform infrared (FTIR) spectroscopy utilizes the infrared region of the electromagnetic spectrum (between 0.8 and 1,000 μm wavelength) to recognize chemical composition of materials. In the case of plastic materials it allows to identify the structural polymer. The infrared spectrum (by transmittance or absorbance) of a sample is recorded by passing a beam of infrared light through the sample. A data-processing technique called Fourier transform converts raw data into the sample's spectrum. Then the sample's spectrum is compared to reference spectra. The samples were cleaned with water and mild detergent, rinsed with deionized water and then dried in oven with air convection at 450 °C for 24 hours.

The characteristics of the instrument utilized in this study for FTIR spectroscopy are:

- FTIR Perkin-Elmer SpectrumOne;
- equipped with HATR, crystal ZnSe, 45°, (pressure 90);
- wavenumber range: 4000-630 cm⁻¹;
- resolution: 4 cm⁻¹;
- number of scanning: 4.

2.2 Methods for waste processing

Waste processing was carried out at laboratory scale to assess the feasibility of recycling. In particular, for the treatment of the different typologies of investigated pharmaceutical waste, comminution operations were evaluated. According to the composition of products (polymeric materials) cutting mills were employed, which apply shearing to reduce particle size. In this study a cutting mill Retsch – SM 2000 equipped with interchangeable sieves to control particle size in output product was utilized to carry out comminution tests.

2.3 Materials

The bottles in HDPE are utilized for suspension to be reconstituted. The analyzed samples are composed by waste bottles, collected at the end of the production line and manually emptied. The bottles are without labels and caps and may contain residual powder. A synthesis of HDPE bottles characteristics is reported in Table 1.
Material HDPE
Longitudinal dimension (cm) 11.0
Transversal dimension (cm) 5.5
average weight (g) 17.4
average powder content (mg) 40.0
average powder content (%) 0.05

Table 1. Synthesis of the characteristics of HDPE bottles.

Material PET
Longitudinal dimension (cm) 14.2
Transversal dimension (cm) 5.2
average weight (g) 23.2
average syrup content (mg) 19.0
average syrup content (%) 8.99

Table 2. Synthesis of the characteristics of PET bottles.

Plastic bags and films derive from the packaging of raw materials utilized in production processes. The analyzed samples are of varying composition and thickness, and contain residual powders, whose composition is in relation to the production cycle. Four different typologies of plastic bags and films were identified:

- white and red bags, containing bicarbonate;
- thin-film;
- bags, with printed character “A” in black;
- bags, with printed character “A” in blue.

The samples were analysed by FTIR spectrometry to identify the polymeric composition (Figure 4).

As resulting from the comparison of acquired spectra with the reference one, all 4 types of materials are polyethylene (PE), in particular the recognized polymeric structure is low-density polyethylene (LDPE).

The residual powders were analysed by dry sieve analysis to identify size distribution, characterized by a mode of the distribution equal to 112 µm, while the top-size is lower than 1,000 µm.

The flexible multi-layered (plastic and aluminium) sachets containing granular medicine are wasted at the end of the production line because of incorrect filling. In this case, the sachets are collected and sent to disposal. The number of wasted sachet is $4 \times 10^6$ per year on average. Table 3 reports a synthesis of the sachets characteristics.
Fig. 4. FTIR spectra of plastic bags and films (y-axis: transmission %; x-axis: wavenumber cm⁻¹): white and red bags, containing bicarbonate (above left), thin-film (above right), bags, with printed character “A” in black (below left), bags, with printed character “A” in blue (below right).

<table>
<thead>
<tr>
<th>Material</th>
<th>plastic and aluminium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Longitudinal dimension (cm)</td>
<td>11.8</td>
</tr>
<tr>
<td>Transversal dimension (cm)</td>
<td>2.2</td>
</tr>
<tr>
<td>average weight of sachet(g)</td>
<td>7.4</td>
</tr>
<tr>
<td>average powder content (mg)</td>
<td>3.2</td>
</tr>
<tr>
<td>average powder content (%)</td>
<td>0.46</td>
</tr>
</tbody>
</table>

Table 3. Synthesis of the characteristics of flexible multi-layered (plastic and aluminium) sachets.

The granular medicine contained in sachets was analysed by dry sieve analysis and by laser granulometer to identify size distribution. Size distribution was analyzed by dry sieve analysis and laser granulometry (Figure 5), showing different mode and top-size. In particular, laser granulometry shows lower value of both (mode: 40 μm, top size: 100 μm) than dry sieve analysis (mode: 280 μm, top size: 1000 μm). This is probably due to the break-up of aggregated granules during mixing in water.

2.4 Preliminary tests

For the recovery of the waste materials, in order to evaluate the possibility of adopting an industrial shredder installed in the production plant under investigation, preliminary comminution tests have been carried out on the following waste typologies:
- primary packaging:
  - bottles in high density polyethylene (HDPE), for suspension to be reconstituted;
  - bottles in poly(ethylene terephthalate) (PET), for syrup;
  - plastic bags and films of varying composition and thickness;
- pharmaceutical waste:
  - flexible multi-layered (plastic and aluminium) sachets containing granular medicine.

![Fig. 5. Size distribution of granular medicine contained in sachets by laser granulometry.](image)

The technical details of the industrial shredder are shown in the Table 4.

<table>
<thead>
<tr>
<th>Producer</th>
<th>Satrind S.p.A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>F615</td>
</tr>
<tr>
<td>Engine power</td>
<td>11 kW</td>
</tr>
<tr>
<td>n. shafts</td>
<td>2</td>
</tr>
<tr>
<td>Speed shafts</td>
<td>19/10-15/8 rpm</td>
</tr>
<tr>
<td>n. blades of 30 mm</td>
<td>19</td>
</tr>
</tbody>
</table>

Table 4. Technical characteristics of industrial shredder installed in the investigated plant.

On the two shafts of the shredder 19 blades are fixed that, thanks mainly to the application of cut stresses, are able to break the waste materials (Figure 6). Some of the material is broken by tear action due to the rotation of the blades. In the preliminary lab tests, the application of cut stresses have been obtained by mean of a blade mill RETSCH - SM 2000, that can be equipped or not with different grids that allow to control the size of the comminuted products. The comminution chamber of the mill is shown in Figure 6.

The preliminary laboratory tests have been conducted in dry conditions adopting two different operational configurations:

- without the grids for the control of the size of the comminuted products;
- with a 20 mm mesh grid.
2.4.1 Results of the preliminary laboratory tests

The preliminary lab tests have shown the effectiveness of the application of cut stresses to break the considered typologies of pharmaceutical waste materials. Moreover, the comminuted products obtained in the tests are characterised by an average lower size that is above the higher average size of the powder and granular medicine contained in the waste sachet (1.0 mm). The results obtained in the preliminary tests are reported for each considered waste typologies in the following.

- **Bottles in high density polyethylene (HDPE), for suspension to be reconstituted**

  The bottles have been divided in two parts in order to reach dimensions suitable for the laboratory blade mill.

  The tests carried out without the adoption of the control grids did not produce useful results, as no breakage were observed in the bottles collected in the output.

  On the contrary, in the tests carried out with the adoption of a 20 mm mesh control grid it was possible to break the bottles in HDPE and reach comminuted products mainly belonging to the size class +1.0 mm. The comminuted products are shown in Figure 7.

- **Bottles in poly(ethylene terephthalate) (PET), for syrup**

  The bottles have been divided in two parts in order to reach dimensions suitable for the laboratory blade mill. The tap and aluminium ring have been kept in the sample. The bottles submitted to the tests did not contain syrup. The tests carried out without the adoption of the control grids did not produce useful results, as no breakage were observed in the bottles collected in the output. On the contrary, in the tests carried out with the adoption of a 20 mm mesh control grid it was possible to break the bottles in PET and reach comminuted products mainly belonging to the size class +1.0 mm. The comminuted products are shown in Figure 7.
Plastic bags and films of varying composition and thickness

The preliminary comminuted tests have been carried out on samples of plastic bags containing bicarbonate and on samples of films. Both sample typologies are made of LDPE.

The plastic bags and films have been cut in samples of 50×50 mm and 100×100 mm in order to reach dimensions suitable for the laboratory blade mill. The tests carried out without the adoption of the control grids did not produce useful results, as no breakage were observed in the samples of 50×50 mm, and clogging and consequent stoppage of the mill for the samples 100×100 mm took place. On the contrary, in the tests carried out with the adoption of a 20 mm mesh control grid it was possible to break the plastic bags and films, both the 50×50 mm and 100×100 mm samples. The comminuted products of 100×100 mm samples of bags and films are shown in Figure 8. When submitted to sieving classification, the comminuted products presented average size generally above 1.0 mm and, therefore, higher than the higher average size of the powder medicine contained in the bags and films.
Flexible multi-layered (plastic and aluminium) sachets containing granular medicine

Preliminary tests were conducted on flexible multi-layered sachets containing granular medicine. The comminution tests resulted effective both without and with the adoption of the 20 mm mesh control grid. Notably, due to the lower resident time, the milling operations conducted without the control grid produced particles belonging to size classes greater than 1.0, i.e. greater than the maximum size of the granulate medicine contained in the sachet. Figure 9 shows the comminuted products classified in the size class +1.0 mm, while Figure 10 and Figure 11 show the comminuted products classified in the size classes obtained -1.0 +0.5 mm and -0.5 mm.

Fig. 9. Comminuted flexible multi-layered sachets containing granular medicine, belonging to the size class +1.0 mm, obtained in the preliminary tests without control grid (left) and with 20 mm mesh control grid (right).

Fig. 10. Comminuted flexible multi-layered sachets containing granular medicine, belonging to the size class -1.0 +0.5 mm, obtained in the preliminary tests without control grid (left) and with 20 mm mesh control grid (right).
2.5 Waste processing (comminution) tests

On the basis of the results of the preliminary tests, the comminution processes in laboratory scale have been set up. Notably, the tests have been carried out adopting the blade mill Retsch - SM 2000 under two operational conditions:

- dry milling, with 2 cm mesh control grid;
- wet milling, with 2 cm mesh control grid.

Wet drying has been realised feeding the mill with the waste materials together with little quantities of water. In such a way, the operational conditions that could be achieved with the shredder installed in the considered industrial plant have been simulated. For the Flexible multi-layered (plastic and aluminium) sachets, tests have been conducted with the blade mill and with a mini-shredder, in order to evaluate the possibility of recovering the granular medicine they contain. The comminuted products have been analysed by mean of:

- dry sieving;
- laser granulometry;
- imagine analysis.

The comminution processes are described in the following paragraphs, for each considered waste typology.

- Bottles in high density polyethylene (HDPE), for suspension to be reconstituted

The HDPE bottles have been cut in their longitudinal axes before feeding them to the blade mill.

In the dry comminution tests, samples made of 5 bottles in HDPE have been adopted. The products of the dry and wet comminution tests have been submitted to dry sieving and image analysis. The sieving tests have been conducted adopting sieves of ASTM series with 2.0 and 1.0 mm mesh. The results of the sieving tests are reported in Figure 12 in terms of cumulative passing for dry and wet comminution tests.
Comparing the results of sieving tests (Figure 13), the dry and wet comminution tests do not show substantial differences in the size distribution of their products.

![Graph showing cumulative passing of particles for dry and wet comminution](image)

**Fig. 12.** Bottles in HDPE, 20 mm grid, cumulative passing, dry (left) and wet (right) comminution.

![Graph comparing particle size distribution for dry and wet comminution](image)

**Fig. 13.** Bottles in HDPE, comparison of the products of dry and wet comminution tests in terms of size distribution.

After the classification of particles in the size classes +2 mm, -2 mm +1 mm, and -1 mm obtained by sieving, image analysis has been conducted on the products of dry and wet comminution tests.

The results of image analysis for the bottles in HDPE are given in Tables 5-10.

Examples of images of the dry and wet comminution products are shown in Figure 14 and Figure 15 respectively.

Comparing the results of the image analysis for the considered size classes, no major differences can be observed in the particle size of the products obtained in the dry and wet comminution tests in terms of the values of the parameters *Area, Major Axis Length, Minor Axis Length* and *Feret Diameter*.

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The high values of the parameter Compactness, measured in the products of both dry and wet comminution products, are in relation with the irregular morphology of particles, reasonably due to the cut stresses applied by the blades of the mill.

The values of the parameter Shape Factor describe a shape of elongated particles.

The statistic parameters, notably the standard deviation, show a high variability in the analysed particles, with standard error and confidence intervals substantially constant for both dry and wet comminution products.

<table>
<thead>
<tr>
<th># Obj</th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
<th>S Factor</th>
<th>Feret D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>205.066</td>
<td>188.244</td>
<td>24.942</td>
<td>13.659</td>
<td>173.927</td>
<td>0.124</td>
<td>15.124</td>
</tr>
<tr>
<td>Min</td>
<td>7.272</td>
<td>34.974</td>
<td>6.584</td>
<td>2.208</td>
<td>27.283</td>
<td>0.009</td>
<td>3.043</td>
</tr>
<tr>
<td>Max</td>
<td>961.603</td>
<td>1437.503</td>
<td>73.747</td>
<td>24.068</td>
<td>753.578</td>
<td>0.359</td>
<td>34.991</td>
</tr>
<tr>
<td>Std Dev</td>
<td>152.176</td>
<td>232.241</td>
<td>10.375</td>
<td>5.290</td>
<td>129.450</td>
<td>0.077</td>
<td>5.712</td>
</tr>
<tr>
<td>Std Err</td>
<td>13.892</td>
<td>21.201</td>
<td>0.947</td>
<td>0.483</td>
<td>11.817</td>
<td>0.007</td>
<td>0.521</td>
</tr>
<tr>
<td>95% Conf</td>
<td>37.879</td>
<td>57.808</td>
<td>2.583</td>
<td>1.317</td>
<td>32.222</td>
<td>0.019</td>
<td>1.422</td>
</tr>
<tr>
<td>99% Conf</td>
<td>49.781</td>
<td>75.973</td>
<td>3.394</td>
<td>1.730</td>
<td>42.347</td>
<td>0.025</td>
<td>1.869</td>
</tr>
</tbody>
</table>

Table 5. Bottles in HDPE, dry comminution, size class: +2.0 mm.
The PET bottles have been fed to the blade mill without any pre-treatment, therefore including the aluminium cap and relative ring, and in some cases also the paper labels. The comminuted tests have been carried out only in dry conditions, as in the wet tests the

- **Bottles in poly(ethylene terephthalate) (PET), for syrup**

Table 6. Bottles in HDPE, dry comminution, size class: -2.0 +1.0 mm.

<table>
<thead>
<tr>
<th># Obj</th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
<th>S Factor</th>
<th>Feret D</th>
</tr>
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<tbody>
<tr>
<td>42</td>
<td></td>
<td>42</td>
<td>42</td>
<td>42</td>
<td>42</td>
<td>42</td>
<td>42</td>
</tr>
<tr>
<td>Mean</td>
<td>5.917</td>
<td>107.792</td>
<td>5.397</td>
<td>2.067</td>
<td>22.963</td>
<td>0.194</td>
<td>2.649</td>
</tr>
<tr>
<td>Min</td>
<td>0.909</td>
<td>22.001</td>
<td>2.209</td>
<td>0.863</td>
<td>9.138</td>
<td>0.018</td>
<td>1.076</td>
</tr>
<tr>
<td>Max</td>
<td>15.461</td>
<td>684.613</td>
<td>11.824</td>
<td>3.209</td>
<td>92.040</td>
<td>0.571</td>
<td>4.437</td>
</tr>
<tr>
<td>Std Dev</td>
<td>3.175</td>
<td>110.433</td>
<td>2.439</td>
<td>0.589</td>
<td>14.100</td>
<td>0.122</td>
<td>0.727</td>
</tr>
<tr>
<td>Std Err</td>
<td>0.490</td>
<td>17.040</td>
<td>0.376</td>
<td>0.091</td>
<td>2.176</td>
<td>0.019</td>
<td>0.112</td>
</tr>
<tr>
<td>95% Conf</td>
<td>0.790</td>
<td>27.488</td>
<td>0.607</td>
<td>0.147</td>
<td>3.510</td>
<td>0.030</td>
<td>0.181</td>
</tr>
<tr>
<td>99% Conf</td>
<td>1.039</td>
<td>36.126</td>
<td>0.798</td>
<td>0.193</td>
<td>4.612</td>
<td>0.040</td>
<td>0.238</td>
</tr>
</tbody>
</table>

Table 7. Bottles in HDPE, dry comminution, size class: -1.0 mm.

<table>
<thead>
<tr>
<th># Obj</th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
<th>S Factor</th>
<th>Feret D</th>
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<td>48</td>
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<td>Mean</td>
<td>1.040</td>
<td>67.968</td>
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<td>0.768</td>
<td>7.582</td>
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<td>Min</td>
<td>0.011</td>
<td>19.554</td>
<td>0.185</td>
<td>0.054</td>
<td>0.514</td>
<td>0.055</td>
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<tr>
<td>Max</td>
<td>3.614</td>
<td>227.840</td>
<td>6.093</td>
<td>2.334</td>
<td>21.682</td>
<td>0.643</td>
<td>2.145</td>
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<tr>
<td>Std Dev</td>
<td>0.930</td>
<td>45.895</td>
<td>1.494</td>
<td>0.433</td>
<td>5.192</td>
<td>0.148</td>
<td>0.532</td>
</tr>
<tr>
<td>Std Err</td>
<td>0.134</td>
<td>6.624</td>
<td>0.216</td>
<td>0.062</td>
<td>0.749</td>
<td>0.021</td>
<td>0.077</td>
</tr>
<tr>
<td>95% Conf</td>
<td>0.232</td>
<td>11.424</td>
<td>0.372</td>
<td>0.108</td>
<td>1.292</td>
<td>0.037</td>
<td>0.132</td>
</tr>
<tr>
<td>99% Conf</td>
<td>0.304</td>
<td>15.014</td>
<td>0.489</td>
<td>0.142</td>
<td>1.698</td>
<td>0.048</td>
<td>0.174</td>
</tr>
</tbody>
</table>

Table 8. Bottles in HDPE, wet comminution, size class: +1 mm.

- Bottles in poly(ethylene terephthalate) (PET), for syrup
products of comminution could not be easily extracted from the mill due to the presence of the syrup acting as a bonding agent for the PET particles and the mill surface. The dry

Fig. 16. Bottles in PET, 20 mm grid, cumulative passing, dry comminution.
comminution tests have been carried out on samples composed of 5 PET bottles. The products of the dry comminution tests have been submitted to dry sieving, laser granulometry and image analysis. The sieving tests have been conducted adopting sieves of ASTM series with 2.0, 1.0 mm and 38 µm mesh. Results of sieving tests and of laser granulometry analysis are reported in Figure 16 and Figure 17, respectively, both as cumulative passing for the dry comminution tests. The size class -38 µm has not been analyzed due to the presence of paper fibres of the labels including fine plastic particles.

After the classification in the particle size classes +2 mm, -2 mm +1 mm, and -1 mm +38 µm obtained by sieving, image analysis have been conducted on the products of dry comminution tests. The results of image analysis for the bottles in PET are given in Tables 11-13. Examples of images of the dry comminution products are shown in Figure 18.

Fig. 17. Bottles in PET, 20 mm grid, size distribution, dry comminution, size class +2.0 – 1.0 mm (left) and -1 mm +38 µm (right), laser granulometry.

Fig. 18. Bottles in PET, images of the products of dry comminution tests.

The high values of the parameter Compactness, measured in the products of both dry comminution products, are in relation with the irregular morphology of particles, reasonably due to the cut stresses applied by the blades of the mill. The values of the parameter Shape Factor describe a shape of elongated particles. The statistic parameters, notably the standard deviation, show a high variability in the analysed particles, in particular for the higher size class (+2 mm), while the intermediate size class (-2 +1 mm) presents more homogeneous values of the morphologic and dimensional parameters.
The composition of the samples that contain the 4 typologies of plastic bags and films used in the dry and wet comminution tests are reported in Table 14.
Weigh (%)  

<table>
<thead>
<tr>
<th>Material typology</th>
<th>Polymer</th>
<th>dry comminution</th>
<th>wet comminution</th>
</tr>
</thead>
<tbody>
<tr>
<td>white and red bags containing bicarbonate</td>
<td>LDPE</td>
<td>23</td>
<td>23</td>
</tr>
<tr>
<td>thin film</td>
<td>LDPE</td>
<td>23</td>
<td>20</td>
</tr>
<tr>
<td>bags, with printed character “A” in black</td>
<td>LDPE</td>
<td>24</td>
<td>27</td>
</tr>
<tr>
<td>bags, with printed character “A” in blue</td>
<td>LDPE</td>
<td>30</td>
<td>30</td>
</tr>
</tbody>
</table>

Table 14. Composition of the samples of plastic bags and films used in the dry and wet comminution tests.

The plastic bags and films have been cut in samples of 50×50 mm in order to reach dimensions suitable for the laboratory blade mill. Moreover, the samples maintained their content of bulk powder.

The products of the dry and wet comminution tests have been submitted to dry sieving, laser granulometry and image analysis.

The sieving tests have been conducted adopting sieves of ASTM series with 2.0 and 1.0 mm mesh. The results of the sieving tests are reported in Figure 19 in terms of cumulative passing for dry and wet comminution tests.

Comparing the results obtained in the sieving tests (Figure 20), the wet comminution shows less particles belonging to the +2 mm size class than the dry comminution.

![Fig. 19. Plastic bags and films, 20 mm grid, cumulative passing, dry (left) and wet (right) comminution.](image-url)

The results of laser granulometric analysis are shown in Figure 21 in terms of size distribution for products of dry and wet comminution tests belonging to the -1 mm size class.
Comparing the results obtained in the laser granulometry analysis, the dry and wet comminution tests do not show substantial differences in the size distribution of their products.

After the division in the particle size classes in +2 mm, -2 mm +1 mm, and -1 mm obtained by sieving, image analysis have been conducted on the products of dry and wet comminution tests. The results of image analysis for the plastic bags and films are given in Tables 15-20. Examples of images taken of the dry and wet comminution products are shown in Figure 22 and Figure 23 respectively.

The results of image analysis are reported in the following for all the considered size classes.

Comparing the results of the image analysis for the considered size classes, the difference between dry and wet comminution tests can be observed in the dimensions of the collected particles, measured by the values of Area, Major Axis Length, Minor Axis Length and Feret Diameter: the analysed particles generally belong to smaller size classes.

The high values of the parameter Compactness, measured in the products of both dry and wet comminution products, are in relation with the irregular morphology of particles, reasonably due to the cut stresses applied by the blades of the mill to very thin material (LDPE).
The values of the parameter Shape Factor describe a shape of elongated particles.

The statistic parameters, notably the standard deviation, show a high variability in the analysed particles, with standard error and confidence intervals substantially constant for both dry and wet comminution products.

<table>
<thead>
<tr>
<th># Obj</th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
<th>S Factor</th>
<th>Feret D</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>84</td>
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<td>84</td>
<td>84</td>
<td>84</td>
<td>84</td>
<td>84</td>
</tr>
<tr>
<td>Mean</td>
<td>110.169</td>
<td>858.617</td>
<td>22.404</td>
<td>11.392</td>
<td>276.048</td>
<td>0.033</td>
<td>10.525</td>
</tr>
<tr>
<td>Min</td>
<td>0.353</td>
<td>45.314</td>
<td>0.943</td>
<td>0.691</td>
<td>3.999</td>
<td>0.003</td>
<td>0.670</td>
</tr>
<tr>
<td>Max</td>
<td>460.604</td>
<td>4994.348</td>
<td>54.142</td>
<td>28.985</td>
<td>1175.889</td>
<td>0.277</td>
<td>24.217</td>
</tr>
<tr>
<td>Std Dev</td>
<td>101.475</td>
<td>828.363</td>
<td>12.335</td>
<td>6.681</td>
<td>232.520</td>
<td>0.038</td>
<td>5.464</td>
</tr>
<tr>
<td>Std Err</td>
<td>11.072</td>
<td>90.382</td>
<td>1.346</td>
<td>0.729</td>
<td>25.370</td>
<td>0.004</td>
<td>0.596</td>
</tr>
<tr>
<td>95% Conf</td>
<td>21.700</td>
<td>177.145</td>
<td>2.638</td>
<td>1.429</td>
<td>49.724</td>
<td>0.008</td>
<td>1.168</td>
</tr>
<tr>
<td>99% Conf</td>
<td>33.195</td>
<td>270.983</td>
<td>4.035</td>
<td>2.186</td>
<td>76.065</td>
<td>0.012</td>
<td>1.787</td>
</tr>
</tbody>
</table>

Table 15. Plastic bags and films, dry comminution, size class: +2 mm.
• **Flexible multi-layered (plastic and aluminium) sachets containing granular medicine.**

The flexible multi-layered sachets have been fed to the blade mill without any pre-treatment, including the granular medicine they contained. For the dry and wet...
comminution tests, samples made of 25 sachets (equal to 184.87 g) and of 10 sachets (equal to 73.70 g) have been respectively used.

<table>
<thead>
<tr>
<th></th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
<th>S Factor</th>
<th>Feret D</th>
</tr>
</thead>
<tbody>
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<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
</tr>
<tr>
<td>Mean</td>
<td>3.638</td>
<td>319.329</td>
<td>4.975</td>
<td>1.915</td>
<td>32.679</td>
<td>0.051</td>
<td>2.039</td>
</tr>
<tr>
<td>Min</td>
<td>0.354</td>
<td>113.617</td>
<td>1.577</td>
<td>0.451</td>
<td>7.857</td>
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<tr>
<td>Max</td>
<td>9.377</td>
<td>776.877</td>
<td>10.951</td>
<td>3.989</td>
<td>83.139</td>
<td>0.111</td>
<td>3.455</td>
</tr>
<tr>
<td>Std Dev</td>
<td>2.309</td>
<td>172.573</td>
<td>2.079</td>
<td>0.847</td>
<td>17.157</td>
<td>0.026</td>
<td>0.695</td>
</tr>
<tr>
<td>Std Err</td>
<td>0.311</td>
<td>23.270</td>
<td>0.280</td>
<td>0.114</td>
<td>2.313</td>
<td>0.003</td>
<td>0.094</td>
</tr>
<tr>
<td>95% Conf</td>
<td>0.575</td>
<td>42.956</td>
<td>0.517</td>
<td>0.211</td>
<td>4.271</td>
<td>0.006</td>
<td>0.173</td>
</tr>
<tr>
<td>99% Conf</td>
<td>0.755</td>
<td>56.454</td>
<td>0.680</td>
<td>0.277</td>
<td>5.613</td>
<td>0.008</td>
<td>0.227</td>
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</table>

Table 19. Plastic bags and films, dry comminution, size class: -2 +1 mm.

<table>
<thead>
<tr>
<th></th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
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<td>75</td>
<td>75</td>
<td>75</td>
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<td>75</td>
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<tr>
<td>Mean</td>
<td>0.679</td>
<td>123.913</td>
<td>2.000</td>
<td>0.809</td>
<td>8.781</td>
<td>0.163</td>
<td>0.851</td>
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<tr>
<td>Min</td>
<td>0.049</td>
<td>16.139</td>
<td>0.463</td>
<td>0.136</td>
<td>1.124</td>
<td>0.027</td>
<td>0.249</td>
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<tr>
<td>Max</td>
<td>3.544</td>
<td>463.666</td>
<td>5.469</td>
<td>2.341</td>
<td>38.710</td>
<td>0.779</td>
<td>2.124</td>
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<tr>
<td>Std Dev</td>
<td>0.606</td>
<td>86.252</td>
<td>1.026</td>
<td>0.527</td>
<td>6.619</td>
<td>0.127</td>
<td>0.377</td>
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<tr>
<td>Std Err</td>
<td>0.070</td>
<td>9.960</td>
<td>0.118</td>
<td>0.061</td>
<td>0.764</td>
<td>0.015</td>
<td>0.044</td>
</tr>
<tr>
<td>95% Conf</td>
<td>0.151</td>
<td>21.470</td>
<td>0.255</td>
<td>0.131</td>
<td>1.648</td>
<td>0.032</td>
<td>0.094</td>
</tr>
<tr>
<td>99% Conf</td>
<td>0.198</td>
<td>28.216</td>
<td>0.336</td>
<td>0.172</td>
<td>2.165</td>
<td>0.041</td>
<td>0.123</td>
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</table>

Table 20. Plastic bags and films, dry comminution, size class: -1 mm.

The products of the dry comminution tests have been submitted to dry sieving, laser granulometry and image analysis.

The sieving tests have been conducted adopting sieves of ASTM series with 1.0 mm, 0.85 mm and 0.50 mm mesh.

The results of the sieving tests are reported in Figure 24 in terms of cumulative passing for the dry comminution tests.

The wet comminution tests have been carried out in order to verify the effect of the use of water on the granular medicine. The results of the tests have been analysed in qualitative terms. In the Figure 25 are shown the images of the products of the comminution test in which 0.4 l of water have been fed to the mill together with the sachets. From the images it can be observed that the sparkling granular medicine has not relevant effects. Moreover, the presence of water allowed reducing the dispersion of powder in the environment during the comminution.
The results of laser granulometry analysis are shown in Figure 26 and Figure 27 in terms of size distribution for products of dry comminution tests belonging to the -1 mm +0.85 mm, -0.85 mm +0.5 mm and -0.5 mm size classes.
The results of the sieving tests show that the comminuted dry sachets are mostly found in the +1.0 mm and -0.5 mm size classes. In fact, in these classes are respectively collected the multi-layered materials and the granular medicine particles. In the size class -1.0 mm +0.85 mm, the results of laser granulometer analyses show two principal modes, reasonably due to the presence of both multi-layered materials and granular medicine.

After the division in the particle size classes in -1 mm +0.85 mm obtained by sieving, image analysis have been conducted on the products of dry comminution tests. The results of image analysis for the multi-layered sachets are given in Table 21. Examples of images taken of the dry comminution products are shown in Figure 28.
The high values of the parameter Compactness, measured in the products of dry comminution products, are in relation with the irregular morphology of multi-layered particles, reasonably due to the cut stresses applied by the blades of the mill.

The values of the parameter Shape Factor describe a shape of elongated particles.

The statistic parameters, notably the standard deviation, show a high variability in the analysed particles, due to the simultaneous presence of multi-layered and granular particles.

<table>
<thead>
<tr>
<th></th>
<th>Area</th>
<th>Compact</th>
<th>Maj Len</th>
<th>Min Len</th>
<th>Perim</th>
<th>S Factor</th>
<th>Feret D</th>
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<td>11</td>
<td>11</td>
<td>11</td>
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<td>11</td>
</tr>
<tr>
<td>Mean</td>
<td>253.133</td>
<td>73.910</td>
<td>27.032</td>
<td>15.394</td>
<td>131.359</td>
<td>0.193</td>
<td>16.894</td>
</tr>
<tr>
<td>Min</td>
<td>32.653</td>
<td>38.744</td>
<td>10.198</td>
<td>3.748</td>
<td>35.569</td>
<td>0.092</td>
<td>6.448</td>
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<tr>
<td>Max</td>
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<td>136.919</td>
<td>41.900</td>
<td>26.437</td>
<td>227.952</td>
<td>0.324</td>
<td>25.123</td>
</tr>
<tr>
<td>Std Dev</td>
<td>154.107</td>
<td>27.727</td>
<td>9.990</td>
<td>7.452</td>
<td>63.551</td>
<td>0.072</td>
<td>6.371</td>
</tr>
<tr>
<td>Std Err</td>
<td>46.465</td>
<td>8.360</td>
<td>3.012</td>
<td>2.247</td>
<td>19.161</td>
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</tr>
<tr>
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<td>38.360</td>
<td>6.902</td>
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<td>1.855</td>
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<td>0.018</td>
<td>1.586</td>
</tr>
<tr>
<td>99% Conf</td>
<td>50.413</td>
<td>9.070</td>
<td>3.268</td>
<td>2.438</td>
<td>20.789</td>
<td>0.024</td>
<td>2.084</td>
</tr>
</tbody>
</table>

Table 21. Flexible multi-layered sachets, dry comminution, size class: -1.0 mm.

3. Conclusions

The results of experimental tests demonstrate the effectiveness of shear stress to comminute primary packaging and waste pharmaceutical product under investigation.

The comminution tests by blade mill RETSCH – SM 2000 show the following outcomes:

- shear stresses on plastic materials determined an irregular and elongated shape on output particles;
- wet and dry conditions are irrelevant on geometric and morphological characteristics of output particles;
- statistical analysis on image analysis data evidenced a high variability in geometric and morphological parameters: this is probably due to plasticity property of materials under investigation and to applied shear stresses;
- size distribution of the plastic particles after comminution is always greater than 1.0 mm and, therefore, greater than powder eventually contained inside packaging (e.g. in pharmaceutical waste).

Considering the outlined results, the comminution process seems to be a feasible treatment for pharmaceutical waste, in order to reduce particle size and to separate packaging materials (mainly plastics) and powder eventually contained.

The wet comminution, even if not influential on geometric and morphological characteristics of output particles, can be adopted to avoid powder dispersion in air.
4. References


The presently common practice of wastes’ land-filling is undesirable due to legislation pressures, rising costs and the poor biodegradability of commonly used materials. Therefore, recycling seems to be the best solution. The purpose of this book is to present the state-of-the-art for the recycling methods of several materials, as well as to propose potential uses of the recycled products. It targets professionals, recycling companies, researchers, academics and graduate students in the fields of waste management and polymer recycling in addition to chemical engineering, mechanical engineering, chemistry and physics. This book comprises 16 chapters covering areas such as, polymer recycling using chemical, thermo-chemical (pyrolysis) or mechanical methods, recycling of waste tires, pharmaceutical packaging and hardwood kraft pulp and potential uses of recycled wastes.

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