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Using factorial experimental design to evaluate the separation of plastics by froth flotation

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ABSTRACT

This paper proposes the use of factorial experimental design as a standard experimental method in the application of froth flotation to plastic separation instead of the commonly used OVAT method (manipulation of one variable at a time). Furthermore, as is common practice in minerals flotation, the parameters of the kinetic model were used as process responses rather than the recovery of plastics in the separation products.

To explain and illustrate the proposed methodology, a set of 32 experimental tests was performed using mixtures of two polymers with approximately the same density, PVC and PS (with mineral charges), with particle size ranging from 2 to 4 mm. The manipulated variables were frother concentration, air flow rate and pH. A three-level full factorial design was conducted. The models establishing the relationships between the manipulated variables and their interactions with the responses (first order kinetic model parameters) were built. The Corrected Akaike Information Criterion was used to select the best fit model and an analysis of variance (ANOVA) was conducted to identify the statistically significant terms of the model.

It was shown that froth flotation can be used to efficiently separate PVC from PS with mineral charges by reducing the floatability of PVC, which largely depends on the action of pH. Within the tested interval, this is the factor that most affects the flotation rate constants.

The results obtained show that the pure error may be of the same magnitude as the sum of squares of the errors, suggesting that there is significant variability within the same experimental conditions. Thus, special care is needed when evaluating and generalizing the process.

1. Introduction

The froth flotation process was first developed to separate minerals and is now widely used in the mineral industry (Wills and Finch, 2016; Kelly and Spottiswood, 1982). Today, it is extensively applied in the separation of polymers of similar density with a view to recycling. Different reagents, pre-treatment methods and flotation conditions have been tested. A number of mechanisms of polymer surface modification and several theories to explain the separation process have been advanced. A comprehensive review on the subject was recently published by Wang et al. (2015a). Since then, the applicability of froth flotation to the separation of different mixtures continues to be extensively studied.

Generally, the published studies on the application of froth flotation to plastic separation use a "one-variable-at-a-time" evaluation/optimization approach. However, froth flotation is a complex multivariable process where interactions between variables can significantly affect responses. Solid phase characteristics such as particle surface chemistry (which relates to the particle's chemistry and prior history – for example, contact with different substances or pre-treatment), particle size and shape can affect the flotation responses. Furthermore, operational variables such as concentration and type of chemical reagents (like frothers and depressants), pulp pH, air flow rate and impeller speed (in the case of mechanical cells) may play a significant role.

In addition to the effect of independent variables on the process responses, interactions may also occur between the variables. Factorial experimental design is a method for planning experiments in which the results undergo statistical analysis to evaluate the significance of the main and interaction effects (see, for example, Sheridan et al., 2002). This method should be used to generate the predictive mathematical models that describe the system's behavior and to determine the optimal operating settings.

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Censori et al. (2016) proposed that the analysis of plastics flotation should be done using the "classical" first order kinetic model commonly used in mineral flotation (Xu, 1998). The present paper proposes that, in plastics flotation, the factorial experimental design uses the kinetic parameters as responses of the process.

The methodology was experimentally demonstrated using a mixture of Polystyrene (PS) with mineral charges and Polyvinyl Chloride (PVC). The separation of plastics by froth flotation has been most extensively studied on the PVC – PET (Polyethylene terephthalate) mixture (see, for example, Abbasi et al., 2010, Burat et al., 2009, Pongstabodee, et al., 2008; Wang et al., 2015b). With some exceptions, such as Wang et al. (2013) and Fraunholcz and Dalmijn (1998), the separation of the PS – PVC mixture is not commonly addressed because, generally, the studies use packaging PS with density slightly above 1.0 g/cm^3. Because PVC density is higher than 1.3 g/cm^3, separating these two polymers by density should not be a problem; therefore, froth flotation is not required. However, PS with mineral charges, which has slightly lower density than PVC, is commonly used in electronic devices (see Martinho et al., 2012). The separation of PVC and PS with mineral charges is required for the production of high-quality products, but, due to their similar density, gravity concentration processes are not effective. Therefore, froth flotation could be an interesting solution, although, as far as the authors know, it has not yet been addressed.

One of the methods used to achieve selectivity in froth flotation is frother concentration modulation, which seems to be one of the simplest methods because this reagent must always be used in the flotation system (Wang et al., 2013). To illustrate the proposed methodology, this study evaluated the separation of PVC from PS with mineral charges by modifying frother concentration, as well as air flow rate and pulp pH.

2. Materials and methods

2.1. Samples and equipment

The 2 different types of plastic samples, PS with mineral charges and PVC, were obtained from speakers and refrigerators, respectively. These were post-consumer items collected at a Portuguese recycling company. To evaluate the efficiency of the process, the selected items had different colors so that the manual sorting of plastics in the products of separation could be done quickly and cheaply. The PS with mineral charges samples were metallic grey and the PVC samples were white.

The particle size range most suited to plastics flotation is 2–6 mm (Wang et al., 2015a, 2015b). This investigation used a 2–4 mm particle size fraction. A narrow size interval was used to minimize any unwanted effects of particle size in the flotation response. The items were shredded in a Retsch SM 2000 cutting mill equipped with a 10 mm aperture screen and the shredder product was classified using a Fritsch Analysette apparatus. After the items were divided using a Jones divider, all the samples used in the flotation tests had equal mass. The sample used in each flotation test consisted of a mixture of 10 g of each plastic type.

The flotation tests were carried out in a single separation stage using a Leeds flotation cell (see Fig. 1) fitted with an additional module to prevent excessive turbulence. The impeller speed used in the experiments was kept at the most constant rate possible, that is, within the 590–630 rpm range. At values lower than about 590 rpm the air was not conveniently dispersed, while at values higher than approximately 630 rpm there was excessive turbulence.

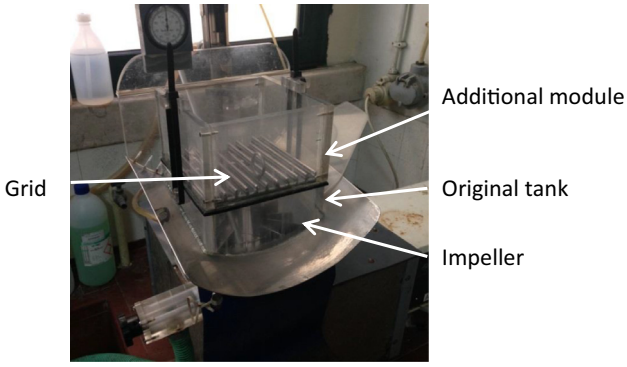

Fig. 1. Leeds flotation cell used in the study.

As shown by Fraunholcz (2004), plastics depression occurs in the presence of cations, thus, distilled water, which is commonly used in mineral flotation studies, should not be used in plastics flotation; therefore, tap water was used instead. The frother used in this study was analytical grade methyl-*iso*-butyl carbinol (MIBC).

2.2. Experimental procedure and factorial design

As mentioned above, frother concentration, air flow rate and pH were the variables chosen for manipulation. The remaining variables were kept constant in all tests. To modify pH, NaOH was added to the solution.

The level (value) of the variables tested in the factorial experimental design (see Table 1) was established in "one-variable-at-a-time" type preliminary tests, conducted with a wide range of variable settings.

The factorial design of the experiments was performed according to the methodology used and comprehensively described by Jordão et al. (2016) in the optimization of the wet shaking process applied to the separation of metals from end-of-life vehicles. The present study used a three-level full factorial design. The number of tests required for the design included all possible combinations of the 3 factors (frother concentration, air flow rate and pH) and of the 3 corresponding levels (high, medium and low) and, as a result, 3^3 experiments were conducted, plus one center point. Five replicates of the center point were used to provide an independent estimate of the experimental error. Thus, 32 ($3^3 + 5$) tests were carried out. All the tests were randomly performed to ensure the independence of observations and errors.

The experimental procedure consisted in: introducing the dry sample in the cell containing water; adjusting the water level; agitating the sample for 10 mins to completely drench the particles; controlling pH at the selected values; adding frother at the selected quantities; agitating for 2 mins, and opening the air valve at the predefined flow rate. Flotation was performed for a total of 26 mins. The concentrate was removed at predefined time intervals of 1, 2, 4, 8, 16, 20, 22, 24 and 26 mins. Both the concentrate and the reject product of each flotation test were dried in an oven for 24 h at 60 °C. They were then allowed to cool to room temperature and weighed.

2.3. Analysis of results

As mentioned before, the process was analyzed using the first order kinetic model parameters (Eq. (1)): PS rate constant (k_{PS}) and ultimate recovery (R_{PS}), PVC rate constant (k_{PVC}) and ultimate recovery (R_{PVC}). The values of these parameters were obtained by

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