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Optimizing a recycling process of SMC composite waste

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Abstract

Investigations about the recycling of sheet moulding compounds (SMC) waste as incorporation material for thermoplastic polymer matrix are reported in this paper. A new efficient process is developed in order to strongly increase the reinforcement glass fraction of SMC leading to good mechanical performance of the new thermoplastic compounds. The overall process is composed of two main steps: mechanical and chemical. The second stage is characterised in terms of optimization and capability by means of experimental design and statistical process control techniques for finding the optimal chemical conditions and validating the process.

1. Introduction

Unsaturated polyester resins are some of the most widely used thermosetting materials in the composites industry (automotive and electrical) (Valette and Hsu, 1999). Commercial interest in glass fibre reinforced polyester composites is principally due to their high strength-to-weight ratio and low cost. Current environmental concerns lead to an increasing development of plastics recycling (Buggy and Farragher, 1995), with the objectives of saving raw materials and also, according to recent European regulations, of limiting the use of landfilling to waste with poor biodegradability. Non recyclable SMC materials widely employed are questioned, a simplified scenario, maybe, but that is the spirit of emerging European waste legislation: Under the guiding concept of extended producer responsibility (EPR), under which manufacturers will be responsible for the environmental impacts of products throughout their life cycles, legislators wish to put the onus for after-life disposal on those in the original supply chain. Moreover, the producer may be responsible not only for the waste itself, but also for risk assessment and for possible harm to humans and the environment (Reinforced Plastics, 2003).

Because the automotive sector is among the largest users of composites, the Brussels legislators have targeted this area first. Their end-of-life vehicles (ELV) directive requires that by 1 January 2015, a minimum of 95% of a vehicle by weight should be re-used or recovered and/or that 85% of it should be re-used or recycled (Reinforced Plastics, 2003, 2001).

For sheet moulding compounds (SMC), the waste grew from 10^8 kg in 1984 to 3.6×10^8 kg in 2000 (Butler, 2001), notably due to end-of-life products. The thermosetting character of SMC coupled with an heterogeneous composition (typically by weight: 20–25 wt% of polyester matrix, 20–25 wt% of glass fibres and 50–55 wt% of calcium carbonate filler) requires the development of new methods of waste management as a matter of valorisation, by example, of one or all parts of each SMC component.

Much of the published work on thermoset recycling discusses polyester-based sheet compositions, for which three main recycling technologies have been considered, involving granulation, incineration and chemical recovery of the polymer matrix (Reiss, 1992; Thermoset Plastics Product Recycling, 1993; Bell, 1993; Collister, 1992; Farrissey et al., 1991). Some steps of these technologies, such as size reduction (Keldermann, 1993; Schaeffer and Plowgan, 1994; Petterson and Nilsson, 1994) and fluidised bed incineration (Kennerly and Kelly, 1998; Pickering and Kelly, 2000; Bream and Hornsby, 2001) preserve the reinforcement

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in a form that can be used again in a new composite, while others concern the recovery of the chemical energy remaining in the resin.

The present work focus on the optimization of a new process allowing the production of obtain ground SMC fractions with characteristics optimised for their introduction as reinforcing fillers in thermoplastic polymers (Pickering and Kelly, 2000; Bream and Hornsby, 2001; Anonymous, 2002; Reygrobellet, 2000; Perrin et al., 2005). In such discontinuous-fibre composites, the performances and properties strongly depend on the properties of the glass fibres (fibre type, volume fraction, fibre aspect ratio, fibre orientation, fibre dispersion and compatibility with the host matrix).

Therefore, the proposed treatment of SMC waste is based on a two-step approach which aims to obtain the longest fibres and the highest fibre content in the final product (about 40%wt). An initial shredding step with a slight crushing (from 2 to 10 mm of average length) (allowing extraction of the fibres from the embedding polyester matrix) is followed by a selective dissolution of calcium carbonate in an acid bath (in order to increase significantly the amount of fibres). The optimization of this process is made by a two-step optimal experimental design approach, followed by a validation of capability by statistical process control. The results of this approach are given in term of process effectiveness, glass fibre length and glass fibre content in the final SMC ground fraction.

2. Experimental

2.1. Materials

SMC composite panels ($500 \times 500 \times 4 \text{ mm}^3$) provided by St Gobain Vetrotex International company were moulded by Compositec SA. Resulting samples constitute model SMC production waste, similar in composition to automotive panels. They contain a relatively low loading (23 wt%) of randomly oriented short E-glass fibres (standard length 4.5 mm), about 55 wt% calcium carbonate and 22 wt% polyester resin.

2.2. Description of the process

As stated in Section 1, the process to optimize consists of two steps (Fig. 1): a slight grinding of the SMC panels followed by a selective dissolution of the ground SMC, in order to finally obtain a filler compound for thermoplastic polymers reinforcement.

Smaller parts of the SMC composites ($50 \times 50 \times 4 \text{ mm}^3$) are first introduced into a rotary cutter mill (Rotoplex-Alpine) equipped with three blades and an out-screen grid 12 mm in diameter. Only one pass was conducted, the residence time being less than 25 min with efficiency equal at about 95% at 1500 rpm. Such conditions allowed us to obtain glass fibres 2–10 mm in length (Perrin et al., 2005) which is comparable to those of commercial glass fibres used for reinforcing thermoplastic polymers.

The dissolution step was conducted in a 5 L Becher vessel with a propeller agitator. This vessel was filled with an acid buffer solution bath in order to selectively dissolve the calcium carbonate present in the ground SMC. The buffer solution consists of 85 vol.% orthophosphoric acid (H_3PO_4) and NaOH. This buffer has a low cost and a low hazardous nature, but it also can be recycled relatively easily (see Section 3.3).

Therefore, in order to obtain an efficient dissolution of the calcium carbonate from the ground SMC, the buffer pH was adjusted to the first pK_a of the triacid (pH 1.9). From the theoretical predominant diagram of CaCO_3 species as a function of pH, we can deduce that in the selected buffer solution (pH 1.9), only the following global dissolution reaction will occur (1):



CO_2 was not a free gas, but the molecule was dissolved in water by controlling the pH, which ensures no impact of global warming.

Also, the main specimens of acid could only be H_3PO_4 and H_2PO_4^- , which does not lead to precipitate in combination with sodium ions from NaOH or calcium ions from CaCO_3 (2–3).

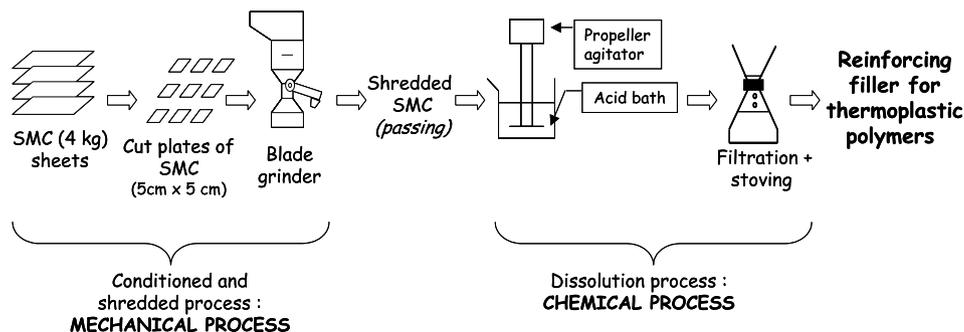
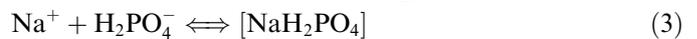


Fig. 1. SMC waste recycling process.

After the dissolution step, the remaining solid fraction (glass fibres, polyester matrix, undissolved CaCO_3) was filtered and dried for 3 h at 80 °C. The filtrate was also reused for further analysis.

2.3. Filler characterisation techniques

Scanning electron microscopy (JEOL JSM 35CF) was used in order to observe the surface of filler particles, and particularly glass fibres.

The composition of filler compounds and of the initial SMC panels was determined as follows:

- Organic fraction, consisting mainly in polyester resin matrix, was first measured by weight loss after calcination at 500 °C for 180 min with a heating rate of 5 °C/min.
- The fraction of CaCO_3 was then measured by calcimetry; this technique consisted of adding hydrochloric acid in excess to about 1–2 g of ground SMC fraction and measuring the volume of CO_2 evolved from the reaction with CaCO_3 ; when experiments were conducted in standard temperature and pressure conditions, the amount of calcium carbonate present in the filler was then given by Eq. (4):

$$\% \text{CaCO}_3 = \frac{100M_{\text{CaCO}_3}V_{\text{CO}_2}}{24040m_{\text{SMC}}} \quad (4)$$

where m_{SMC} is the mass of SMC introduced, V_{CO_2} is the volume of CO_2 evolved and M_{CaCO_3} is the molar mass of CaCO_3 ,

- The remaining glass fibre fraction was calculated by mass balance.

3. Process optimization method

The optimization was focused on the second step of the process (selective dissolution of CaCO_3), and was divided into three parts:

- Optimal experimental design approach.
- Validation of the designed process by the Statistical Process Control method.
- Possibility of reusing the filtrate after the process, as well as its recyclability.

The optimal experimental design was carried out using a two-step approach: a quick identification of the main factors using a simple screening experiment, followed by the application of a complex response surface design for further optimization. All of the experiments were carried out independently and repeated four times.

3.1. Screening of physico-chemical parameters

The effects of four factors known to influence dissolution conditions, sample mass, rotation speed, agitation

time and concentration, were studied using a statistical approach. All of the variables were investigated at two widely spaced levels. The complete matrix for screening was designed using a standard Plackett-Burman orthogonal array. A set of 16 experiments was carried out to determine dissolution conditions under different combinations (Table 1). A factorial type linear model for the answer Y (% CaCO_3) is given by Eq. (5):

$$Y = a_0 + \sum_{i=1}^n a_i X_i + \varepsilon, n = 4 \quad (5)$$

The statistical software package Nemrodw[®] version 2000, LPRAI (Marseille, France) was used to analyse the experimental design.

3.2. Optimization of key determinants

The two key determinants identified (solution concentration [C] and agitation time [T], see Section 4 for details) were optimized for maximizing the dissolution of CaCO_3 with a steady pH 1.9 and 100 g of ground SMC. This result will have a direct impact on the amount of fibres and polyester, and its maximal dissolution will allow supplementary mechanical properties on ground SMC into thermoplastics to be maintained. The response surface approach involving a face-central composite design (FCCCD) was adopted for optimizing the calcium carbonate dissolution conditions. A set of nine experiments including one centre point was carried out. Each variable was studied at three different levels (−1, 0, +1). All variables were taken at a central coded value of zero. The minimum and maximum range of variables investigated and the full experimental plan with respect to their actual and coded forms are listed in Table 2. A multiple regression analysis of the data was carried out to obtain an empirical model defining the response Y , which is the percentage of calcium carbonate dissolved,

Table 1
Plackett-Burman experimental plan for screening of important determinants of dissolution conditions

Variable ^a	High level (+1)	Low level (−1)
	100	20
Rotative speed (rpm)	1100	600
Agitation period (min)	60	30
Concentration of solution ^b (mol L ^{−1})	0.30	0.15

^a The variables are set at two widely spaced levels to separate significantly the effect.

^b The acid used is orthophosphoric acid at pH = $\text{p}K_a = 1.9$.

Table 2
Experimental range of the two variables studied using FCCCD in terms of actual and coded factors

Variable	Range of variables		
	Low (−1)	Mid (0)	High (+1)
Concentration (mol l ^{−1})	0.20	0.40	0.60
Agitation length (min)	30	60	90

in terms of the independent variables. The response surface graphs were obtained using the Nemrodw software to determine:

- the effect of the variables individually and in combination, and
- the optimum level for maximal dissolution conditions.

A factorial type linear model for which the answer Y (%CaCO₃) was given by Eq. (6).

$$Y = a_0 + \sum_{i=1}^n a_i X_i + \sum_{i=1}^{n-1} \sum_{j=i+1}^n a_{ij} X_i X_j + \sum_{i=1}^n a_i^2 X_i + \varepsilon, \quad n = 2 \quad (6)$$

3.3. Reuse of filtrate

A recycling of the filtrate can be expected in order to reuse the phosphorus solution. Consecutive dissolutions of SMC into the same buffered orthophosphoric acid were carried out. The filtrate was reused after adding of a small amount of acid to keep the pH constant at 1.9. The precise volume of added acid after each series has been determined. The process is illustrated on Fig. 2.

3.4. Validation of the designed process by the statistical process control method

Statistical process control (SPC) is a collection of powerful problem-solving techniques which are useful in achieving process stability, as well as improving capability through the reduction of variability, and can be used in any process (Montgomery, 1996). The final objective is to validate a process by the determination of lower and upper control limits and process capability indices. In our present work, this method was applied to the dissolution of calcium carbonate to maximize the fibre content. The statistical model was validated with respect to the two optimized variables (solution concentration $[C]$ and agitation period $[T]$); 16 measurements of calcium carbonate concentration and then of glass fibres rate were carried out and repeated four times. Basic Shewart's chart was used to identify the status of the obtained data to determine if they are in statistical control or not. Mean-chart was only constructed for monitoring process variance. The validation of the dissolution process was described in terms of process capability indices C_p and C_{p_k} (see calculi Eqs. (9) and (10)) conditioned by UCL (upper control limit) and LCL (lower con-

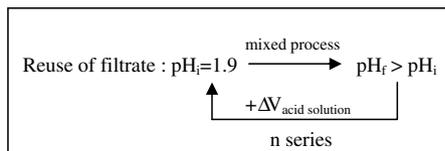


Fig. 2. Reuse of buffered acid filtrate solution.

trol limit) located around a CL (central line), which is the sample grand mean (average of averages). They were calculated following Eqs. (7), (8). The indices of capability make it possible to know if one is able to deliver the product in the required range and to prove, by the use of a policy of quality, improvement of the characteristics of the delivered product.

$$CL = \bar{\bar{X}} \quad (7)$$

$$UCL = CL + A\sigma \text{ and } LCL = CL - A\sigma \quad (8)$$

where $\bar{\bar{X}} = \frac{1}{m} \sum_{j=1}^m \bar{X}_j$ with $\bar{X} = \frac{1}{n} \sum_{i=1}^n x_{ij}$ ($n = 4$ tests and $m = 16$ experiments), $A = 3/\sqrt{m}$ and $\sigma = \sqrt{\frac{1}{m(n-1)} \sum_{j=1}^m \sum_{i=1}^n (x_{ij} - \bar{X})^2}$.

Based on the new control charts (no out-of-control conditions or only common causes appear), process capability studies were conducted and process capability indices (C_p and C_{p_k}) were computed according to Eqs. (9) and (10).

$$C_{p_k} = \min(C_{p_u}, C_{p_l}) \text{ with } C_{p_k} \geq 1.33 \quad (9)$$

$$C_p = \frac{USL - LSL}{6\sigma} \text{ with } C_p \geq 1.33 \quad (10)$$

with $C_{p_u} = (USL - CL)/3\sigma$ and $C_{p_l} = (CL - LSL)/3\sigma$. LSL and USL standing for upper specification limit and lower specification limit, respectively, 40 and 43 wt% of glass fibre content.

Process capability generally refers to whether or not a process is operated in accordance with the specification. If $C_p < 1$, the process is out the specification limits and is not capable of producing in-specification products. C_p can be higher than 1, but bad fibre percentages could still be obtained if the process is not centred. Therefore, C_{p_k} corresponds to the capability index in which the process centre is considered. If C_{p_k} is higher than 1, then 99.73% of the products in the process will be within specification limits. Generally, C_p and C_{p_k} values of more than 1.33 are required by most modern industrial manufacturers (mass production) (Pitt, 1996; Ipek et al., 1999). Consequently, this study will consider a minimum value of 1.33.

About the evaluation of the process with control charts, the following points are considered as important (Srikaeo et al., 2005):

- Two-thirds of the points have to be on the central line or near the central line.
- Only a minimum of points has to be near the control limits.
- The points should randomly fall below or above central limits and should be balanced; and
- There should be no points out-of-control limits.

4. Results and discussion

The aspect and the composition after shredding of the ground SMC before the dissolution step are presented in Fig. 3 and Table 3. Observation of the comminuted recyc-



Fig. 3. Appearance of shredded SMC.

Table 3
Composition and fibre lengths of SMC before and after shredding

Main components of SMC composite	Weight fraction (%) before shredding	Weight fraction (%) after shredding
Polyester resin	25 ± 2	23 ± 2
Calcium carbonate	50 ± 2	51 ± 2
E-glass fibre	25 ± 2 (25–50 mm)	26 ± 2 (2–10 mm)

late micrographs revealed that the recycle particulates are covered by a thin layer of fine powder, electrostatically adhering to their surface (Bream and Hornsby, 2001; Perrin et al., 2005).

Furthermore, particle size analysis of recycle revealed large particles: 20% of the recycle is larger than 10 mm and the greatest part ranges between 2 and 10 mm. These particles may exert a significant influence on the performance of the recycle in the thermoplastic composites. Particularly, it is well known that the stress concentrating effect of larger particles (about micron scale) can lead to a reduction in strength (Beldzki and Kurek, 1994; Jutte and Graham, 1991).

4.1. Optimization of dissolution conditions

The screening study highlighted the role of concentration and agitation length on calcium carbonate dissolution from ground SMC. As seen in Fig. 4, the rotation speed clearly does not favour the increase or decrease of the CaCO_3 dissolution. In contrast, ground SMC weight, agitation length and solution concentration are crucial parameters. Nevertheless, in order to highlight the influence

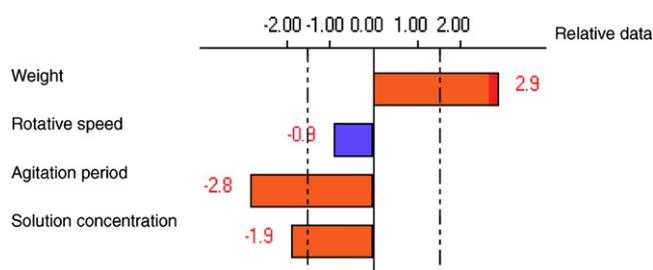


Fig. 4. Graphic study of positive or negative factors influence about CaCO_3 dissolution.

of the agitation length and the solution concentration, the ground SMC weight was fixed at 100 g for further optimization of the two other parameters. Besides, the rotative speed was fixed to 1100 rpm.

4.2. Optimization of the key determinants of dissolution conditions

The Nemrodw[®] analysis of the optimisation study showed that the model terms Z_1 , Z_2 , Z_1^2 , Z_2^2 are significant model terms (the normal plot “Henry’s plot” is found as a straight line evolution). The coefficient of regression equation was calculated using Nemrodw[®] and the data was fitted to a second order polynomial equation. The calcite dissolution conditions can be expressed in terms of the following regression Eq. (11):

$$Y = 20.144 - 7.360Z_1 - 1.754Z_2 + 3.433Z_1^2 + 0.133Z_2^2 \quad (11)$$

with $Z_1 = 0.977X_1 + 0.213X_2$ and $Z_2 = -0.213X_1 + 0.977X_2$ where X_1 is the solution concentration and X_2 is the agitation length.

The regression equation obtained from analysis of variance indicated that the R^2 value (multiple correlation coefficients) is 0.994 (a value higher than 0.75 leads to the acceptance of the model). The “adjusted R^2 ” and “predicted R^2 ” values are 0.984 and 0.929, respectively. For a good statistical model, the R^2 value should be positive and close to 1.0 and all three factors ought to be positive and close to each other, as it was the case here. This suggests that the model can be used in the whole design space. Also, the model indicates that the standard deviation forecast a value of 2.87, which confirms a satisfactory adjustment of the reduced quadratic model to the experimental data.

So as to obtain the best mechanical properties with SMC fillers incorporated into thermoplastic polymers, a ratio of 40–43 wt% fibre content had to be the optimal value. It is also estimated that a final calcite amount of 24–27 wt% would be a good fit for obtaining the ideal fibres ratio (Perrin et al., 2005).

The 2-D and 3-D response surface curves (Fig. 5) were used to determine the optimum level of each variable for an optimised 24–27 wt% calcite ratio. The left figure (Fig. 5a) has revealed a range of calcite ratio from 24 to 27 wt%. Due to process implementation opportunities and according to the response surface, an acid concentration C of 0.30 mol l^{-1} (25 wt% of concentration range) and an agitation period T of 60 min (50 wt% of the agitation period) were selected. The right figure (Fig. 5b) has predicted a reduced quadratic evolution of the calcite dissolution given by the chemical Eq. (1) according to the model.

4.3. Validation of the experimental design

The model was validated for the two variables within the design space. A random set of six calcite dissolution

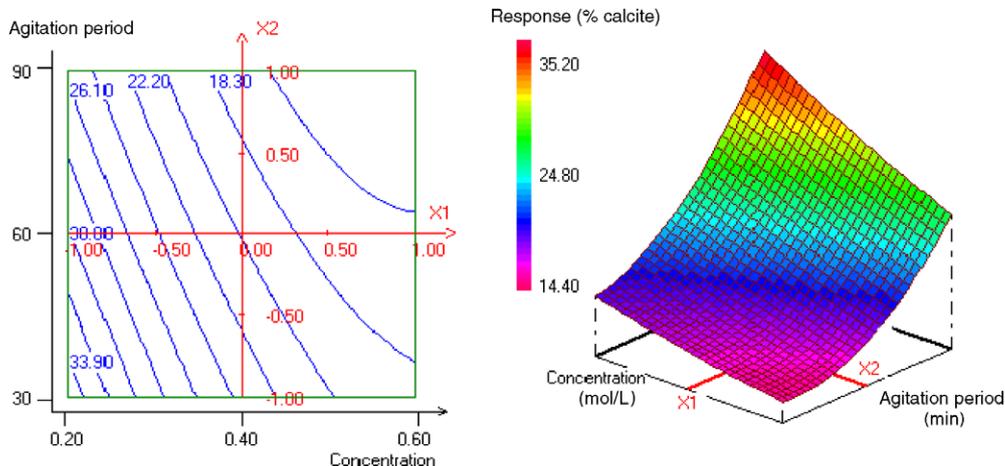


Fig. 5. Response surface curve of calcite dissolution (% CaCO_3) from the 100 g-ground SMCs as a function of buffered acid concentration and agitation period under optimal conditions of pH 1.9-orthophosphoric acid.

experiments, three times repeated, was carried out and compared with the statistical model for calcite ratio (Table 4).

The experimentally determined production dissolution values were in close agreement with the statistically predicted ones, confirming the model's validation. The optimisation dissolution conditions were a concentration of 0.30 mol/L buffered orthophosphoric acid value, 60 min for a 100 g sample of ground SMC dissolved into the pH 1.9 solution.

4.4. Reuse of filtrate

Results of the 10-series test of solubility are presented on Table 5. The analysis method is based on the acid base reaction between the buffer acid bath, which is regenerated on each cycle so as to keep steady the initial pH of the solution. It is noticed that the pH range before the initial and final pH was roughly equivalent for each test. The presence of insoluble particles is never observed. Consequently, it appears very interesting for industrial applications to recycle at least 10 times the whole buffer acid bath without changing the filtrate solution and without losses. It is noticed that this filtrate, based on phosphoric acid, could be precipitated in phosphate to give apatite (Ca_3PO_4) in order to be used as artificial teeth or fertilizer.

Table 4
Validation of reduced quadratic model within the design space

Series No.	Buffered acid concentration (mol/L)	Agitation period (min)	Initial pH	Ground SMC weight (g)	Calcite amount (calcite dissolution) (%)		
					Predicted	Experimental	Model efficiency (standard deviation)
1	0.28	76	1.9	100	23.17	22.84	0.23
2	0.30	40	1.9	100	27.23	28.82	1.12
3	0.47	70	1.9	100	17.16	17.10	0.04
4	0.52	39	1.9	100	19.04	18.80	0.17
5	0.38	65	1.9	100	20.25	20.29	0.02
6	0.21	33	1.9	100	34.16	34.09	0.05

4.5. Statistical process control study: determination of the process capability

The X evolution is carried out by the estimate of the process capability. The results of Shewart's card control from the mixture process is presented on Fig. 6 and obtained from Eqs. (7),(10). The average of average values CL indicates a glass fibres percentage of 41.4 wt% and the standard deviation σ is 0.29. The upper control limit UCL equals

Table 5
Influence of the series number on the solubility

Series	Initial pH	Final pH	ΔV_{sol} (%)	Δ (added acid volume) (%)	Solubility
0	1.9	2.3	5.0	–	Soluble
1	1.9	2.2	4.5	36	Soluble
2	1.9	2.4	6.2	50	Soluble
3	1.9	2.3	5.2	37	Soluble
4	1.9	2.3	5.5	44	Soluble
5	1.9	2.3	4.9	42	Soluble
6	1.9	2.1	4.1	32	Soluble
7	1.9	2.3	5.0	35	Soluble
8	1.9	2.2	4.3	34	Soluble
9	1.9	2.2	4.6	31	Soluble
10	1.9	2.3	4.8	37	Soluble

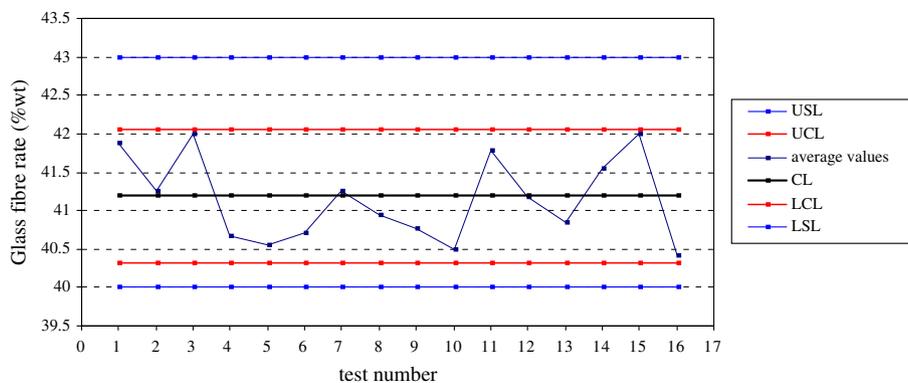


Fig. 6. Shewhart's card control of shredding-dissolution process of SMC.

42.1 wt% and the lower control limit LCL is 40.3 wt%. The analysis of the results reveals process capability indices Cp_k and Cp of 1.37 and 1.72, respectively. These values show a rather good capability because both indices are higher than 1.33.

The rules of control cards were then entirely respected giving a well centered and capable process. The dispersion of test points was relatively low. Moreover, all of the points are well scattered between the average values UCL and LCL providing a confirmation of the good capability.

4.6. Final process of calcium carbonate dissolution

The whole process of calcium carbonate dissolution allowing a strong increase of the fraction of reinforcing fibres or fillers has already been described in Fig. 1.

The target has been reached by the modification of a SMC having a glass fibres fraction of about 41 wt% versus initially only 25 wt%. The new process consists of two main stages: mechanical and chemical. In the first one, only a slight grinding is made with SMC plates conditioned in lumps of 25 cm² squares; 100 g of passing SMC fraction is then put into a 5 L-buffered orthophosphoric acid solution bath with a concentration of 0.3 mol l⁻¹ for an hour and the carbonate calcium part is dissolved as controlled by the design of the experiments. In fact, almost 50% of the CaCO₃ is dissolved, giving more than 40 wt% of glass fibres fraction. Finally, after filtration and stoving, reinforcing filler for thermoplastic polymers is obtained with a yield of 70% with respect to the initial weight of SMC.

5. Conclusion

A modified SMC composite material for incorporation into thermoplastics in order to improve their mechanical properties was successfully developed using a physicochemical process from SMC plates. The design of the process has been investigated in terms of optimization of different parameters using an experimental design and in terms of capability by statistical process control techniques. Resulting data highlighted the main influential factors which were

acid concentration and agitation period. Moreover, successive reuses of the filtrate solution in the dissolution process can be achieved. The dissolution process was studied using SPC, and this has concluded to a rather acceptable capability because Cp_k and Cp values are higher than 1.33.

Finally, a SMC able to be incorporated into thermoplastics with a rate more than 40 wt% of glass fibres instead of only 25 wt% initially, was prepared. This rise offers the possibility to this reused material to compete with new glass fibres provided the use of relevant surface treatments.

It is important to notice that a scale-up has been realised successfully on a quantity of SMC wastes, from automotive bonnets and boots, bordering the 200 kg batches. Moreover, the complete process of recycling has been just rewarded by the Prize from the Innovating Techniques 2006 waste category decreed by ADEME (French national agency for the environment).

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