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Multiobjective optimization of an emulsion copolymerization process

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Abstract: A multiobjective optimization procedure based on evolutionary algorithm has been developed to determine the optimal control policies for a fed-batch emulsion copolymerization reactor, particularly for styrene and butyl acrylate in the presence of n-C12 mercaptan as chain transfer agent (CTA). The process model was elaborated and validated experimentally in order to predict the global monomer conversion, the number and weight average molecular weights, the particle size distribution and the residual monomers. The process objectives are to produce core-shell particles with specific end-use properties and high productivity. This has been achieved by the maximization of the conversion at the end of the process and the minimization of the error between the glass transition temperature and a designed profile subject to a set of operational constraints. The nondominated Pareto solutions obtained were ranked according to the decision maker preferences using multiple attribute utility theory (MAUT). The selected solution gives the best set of the decision variables to be implemented to the real system.

Keywords: multiobjective optimization, core-shell particles, decision aid

1. INTRODUCTION

Multiobjective optimization problems are encountered in most real-world applications and more recently in chemical processes (Fonteix et al. (2004), Garg and Gupta (1999), Mitra et al. (2004), Sakar et al. (2007)). Since such problems involve several objective functions with conflicting nature, the final optimum is not unique but a set of non dominated solutions (the Pareto front) which show a trade-off between the whole objectives. Genetic algorithms (GAs) are well adapted tools to solve multiobjective problems. This kind of technique stands for a class of stochastic optimization methods that simulate the process of natural evolution (mainly genetic algorithms, evolutionary programming, and evolution strategies). These algorithms have proven themselves as a general, robust and powerful search mechanism. Moreover, Evolutionary algorithms (EAs) seem to be especially suited to multiobjective optimization because they are able to find multiple Pareto-optimal solutions in a single simulation run.
Emulsion polymerization is an important industrial process used to produce a great variety of polymers of multiple uses (e.g. paints, adhesives, coatings, varnishes). Moreover, it has significant advantages over bulk and solution polymerization processes such as heat removal capacity and viscosity control. These advantages result mostly from the multiphase and compartmentalized nature of the emulsion polymerization which allows the production of polymers of high molecular weight at high polymerization rates,

delivering a high versatility to product qualities. However, the complexity of emulsion polymerization systems arising from factors such as the multiphase nature, nonlinear behaviour and sensitivity to disturbances induce more intense difficulties on modelling and make the development of optimization procedures of emulsion polymerization reactions a very challenging task.
The end-use properties of the products obtained by emulsion polymerization and copolymerization are governed by the molecular weight distribution (MWD), polymer microstructure, glass transition temperature (T_g), particle size distribution (PSD) and particles morphology. These parameters must be involved in the process design, optimization and control in order to produce latex particles with specific and controlled properties.
The present paper deals with a multiobjective dynamic optimization of an emulsion copolymerization fed-batch reactor. The aim is to produce core-shell particles with specific mechanical and film-forming properties with high productivity. These characteristics are achieved by using two objective functions subject to a set of tight operational constraints and the mathematical model of the system. The first objective function is related to the glass transition temperature of both core and shell while the second deals with the final conversion.
The nondominated solutions (Pareto's front) are obtained by using evolutionary algorithm (EA). This set of optimal solution is ranked according to the decision maker preferences by using multiattribute utility theory (MAUT)

which leads to the selection of the unique solution to be implemented.

2. MULTIOBJECTIVE OPTIMIZATION

A multiobjective optimization problem (MOP) involves simultaneous optimization of more than one objective function. That is the case of the most real-life optimization problems faced in industry which usually deal with more than one competing objective. Traditionally, such problems are solved taking a weighted average of all of the objectives and treating it as a single objective optimization problem. However, the solution then depends on the chosen weights, which, in turn, are subject to individual perception and knowledge of the process. This is quite arbitrary, and a deficiency is always inherent in this method. Single and multiple objective function optimization problems are therefore conceptually different. In multiobjective optimization, there is no optimal solution that could be characterized as the best solution (i.e. global optimum) with respect to all objective functions. Instead, there is an entire set of solutions that are equally good. The solutions are known as Pareto-optimal solutions (i.e. non-dominated solutions). The values of the objective functions corresponding to the set of optimal solutions are called the Pareto front and represent the best trade-offs between the considered often conflicting objectives (Fig.1).

A Pareto-optimal set provides a wide range of design and operational options to designers and practitioners and, hence, enhances the possibility of finding more efficient processes.
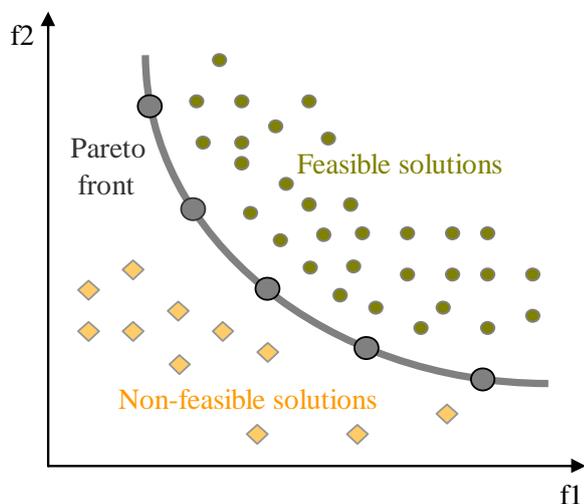
Fig. 1. Illustration of Pareto optimal set (Min (f_1, f_2))

Population-based algorithms such genetic algorithms (GAs) have proven there efficiency to find Pareto-optimal set (Gupta and Gupta (1998), Silva and Biscaia (2003), Mitra et al. (2004)). GA is a search technique based on the working principles of genetics and natural selection; it employs a population-based approach whereby the search for a solution is performed with a group of estimated solutions rather than a single one. Starting with a set of randomly generated initial estimates of the decision variables, GA tries to reach the solution with the help of special operators. In each generation, new set of values of the decision

variables are found through special operations, namely, reproduction, crossover, and mutation, on their old values. This is done in an attempt to produce more desirable objective values until a preassigned number of generations is computed or a limiting condition is reached.

2.1 Process model

The mathematical model for the present study is based on the kinetics of the complex elementary chemical reactions occurring both in the aqueous phase and in the particles, the particle nucleation, radical absorption and desorption (Table 1).

Aqueous phase	
Initiation	$I_2 \xrightarrow{k_d} 2R_{aq}^\bullet$
Inihibition	$R_{aq}^\bullet + Z_{aq} \xrightarrow{k_{zaq}} P + Z_{aq}^\bullet$
Nucleation	$R_{aq}^\bullet + micelle \xrightarrow{k_N} particle + R^\bullet$
Radical absorption	$R_{aq}^\bullet + particle \xrightarrow{k_{cp}} particle + R^\bullet$
Organic phase	
Propagation	$R_i^\bullet + M_j \xrightarrow{k_{pij}} R_j^\bullet$
Termination by combination	$R_i^\bullet + R_j^\bullet \xrightarrow{k_{tcij}} P$
Termination by disproportionation	$R_i^\bullet + R_j^\bullet \xrightarrow{k_{tdij}} 2P$
Inihibition	$R_i^\bullet + Z_p \xrightarrow{k_{zp}} P + Z_p^\bullet$
Transfer to monomers	$R_i^\bullet + M_j \xrightarrow{k_{trmij}} P + R_j^\bullet$
Transfer, chain transfer agent-monomers	$R_i^\bullet + TA_p \xrightarrow{k_{TAp}} P + TA_p^\bullet$
Radical desorption	$R^\bullet \xrightarrow{k_{des}} R_{aq}^\bullet$

Table 1. Kinetic scheme for emulsion copolymerization $(i, j = 1, 2)$

The reaction rates derived from the kinetic scheme, the species partition, the gel and glass effects and the effect of the temperature are not presented here for the sake of brevity.

The process model of the emulsion copolymerization of styrene and butyl acrylate in the presence of n-C12 mercaptan as chain transfer agent (CTA) was developed and validated experimentally for a batch reactor and extended to the fed-batch case. The objective of the model is to predict different variables including overall monomers conversion, number and weight average molecular weights, particle size distribution and residual monomer fractions. The model obtained is a hybrid nonlinear system presented in (Table 2). As mentioned before, for brevity reasons the significations of the different terms are not presented here and the sketch of the model stands to show the complexity of the system and the different phenomena involved in the process. More details of the novelties and the approach used to elaborate the model are presented in Benyahia et al. (2008).

$$\frac{dV_R}{dt} = Q_f + Q_{If} + \sum_{i=1,2} \left(\frac{1}{\rho_{pi}} - \frac{1}{\rho_i} \right) M_M^i \left(\mathcal{R}_{pi} + \mathcal{R}_{trmi} \right)$$

$$\frac{dM_i}{dt} = -\mathcal{R}_{pi} - \mathcal{R}_{trmi} + Q_f [M_i]_f$$

$$\frac{dM_{Ti}}{dt} = Q_f [M_i]_f$$

$$\frac{dI}{dt} = -\mathcal{R}_d + Q_{If} [I]_f$$

$$\frac{dZ}{dt} = -\left(\mathcal{R}_{Zp1} + \mathcal{R}_{Zp2} \right) + Q_f [Z]_f$$

$$\frac{dTA}{dt} = -\mathcal{R}_{TAp1} - \mathcal{R}_{TAp2} + Q_f [TA]_f$$

$$\frac{dS}{dt} = Q_f [S]_f$$

$$\frac{dN_p}{dt} = \mathcal{R}_N$$

$$\frac{d(N_p \bar{n})}{dt} = \mathcal{R}_N + \mathcal{R}_{cp} - \left(\mathcal{R}_{Zp} + \mathcal{R}_T + \mathcal{R}_{des} \right)$$

$$\frac{d(N_P \tilde{n})}{dt} = 2\mathcal{R}_{cp}\bar{n} - \left(\frac{2\tilde{n}}{\bar{n}} + 1 \right) \mathcal{R}_T - 2\frac{\tilde{n}}{\bar{n}} \left(\mathcal{R}_{des} + \mathcal{R}_{TAp} \right)$$

$$\frac{dR_1}{dt} = (\mathcal{R}_N + \mathcal{R}_{cp}) f_{aq1} - \mathcal{R}_{p12} + \mathcal{R}_{p21} - \mathcal{R}_{trm12}$$
$$\qquad + \mathcal{R}_{trm21} - \mathcal{R}_{Zp1} - \mathcal{R}_{des1} - (\mathcal{R}_{T11} + \mathcal{R}_{T12})$$

$$\frac{dR_2}{dt} = (\mathcal{R}_N + \mathcal{R}_{cp}) f_{aq2} - \mathcal{R}_{p21} + \mathcal{R}_{p12} - \mathcal{R}_{trm21}$$
$$\qquad + \mathcal{R}_{trm12} - \mathcal{R}_{Zp2} - \mathcal{R}_{des2} - (\mathcal{R}_{T22} + \mathcal{R}_{T21})$$

$$\frac{d(N_P \bar{n} \chi_1)}{dt} = (\mathcal{R}_N + \mathcal{R}_{cp}) f_{aq1} + \mathcal{R}_{trm21} + \mathcal{R}_{trm11}$$
$$\qquad + \mathcal{R}_{TAp1} - \mathcal{R}_{des1} - \left(\mathcal{R}_{trm11} + \mathcal{R}_{trm12} \right.$$
$$\qquad + \mathcal{R}_{p11} + \mathcal{R}_{p12} + \mathcal{R}_{TAp1} + \mathcal{R}_{Zp1} \big) \chi_1$$
$$\qquad - (\mathcal{R}_{T11} + \mathcal{R}_{T12}) \chi_1$$

$$\frac{d(N_P \bar{n} \chi_2)}{dt} = (\mathcal{R}_N + \mathcal{R}_{cp}) f_{aq2} + \mathcal{R}_{trm12} + \mathcal{R}_{trm22}$$
$$\qquad + \mathcal{R}_{TAp2} - \mathcal{R}_{des2} - \left(\mathcal{R}_{trm22} + \mathcal{R}_{trm21} \right.$$
$$\qquad + \mathcal{R}_{p22} + \mathcal{R}_{p21} + \mathcal{R}_{TAp2} + \mathcal{R}_{Zp2} \big) \chi_2$$
$$\qquad - (\mathcal{R}_{T22} + \mathcal{R}_{T21}) \chi_2$$

$$\frac{d(N_p \bar{n} \lambda_1)}{dt} = \mathcal{R}_N + \mathcal{R}_{cp} - \mathcal{R}_{des} + \mathcal{R}_p + \left(\mathcal{R}_{trm} + \mathcal{R}_{TAp} \right)$$
$$\qquad (1 - \lambda_1) - \left(\mathcal{R}_{Zp} + \mathcal{R}_T \right) \lambda_1$$

$$\frac{d(N_p \bar{n} \lambda_2)}{dt} = \mathcal{R}_N + \mathcal{R}_{cp} - \mathcal{R}_{des} + \mathcal{R}_p (1 + 2\lambda_1)$$
$$\qquad + \left(\mathcal{R}_{trm} + \mathcal{R}_{TAp} \right) (1 - \lambda_2) - \left(\mathcal{R}_{Zp} + \mathcal{R}_T \right) \lambda_2$$

$$\frac{d(N_m)}{dt} = \mathcal{R}_{Zp} + \mathcal{R}_{trm} + \mathcal{R}_{TD} + \mathcal{R}_{TAp} + \frac{\mathcal{R}_{TC}}{2}$$

$$\frac{d(N_m L_1)}{dt} = \lambda_1 \left(\mathcal{R}_{Zp} + \mathcal{R}_{trm} + \mathcal{R}_{TD} + \mathcal{R}_{TAp} + \mathcal{R}_{TC} \right)$$

$$\frac{d(N_m L_2)}{dt} = \lambda_2 \left(\mathcal{R}_{Zp} + \mathcal{R}_{trm} + \mathcal{R}_{TD} + \mathcal{R}_{TAp} \right)$$
$$\qquad + \mathcal{R}_{TC} \left(\lambda_2 + \lambda_1^2 \right)$$

Table 2. Process model equations

(a)

(b)
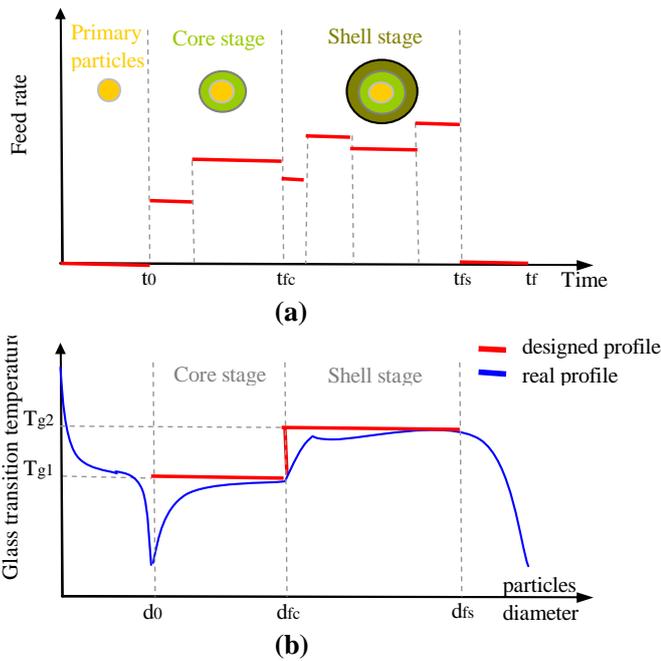
Fig. 2. (a) Feed rate profile (b) Glass transition temperature profile

2.2 Formulation of the problem

The objective of the process is to produce core-shell particles with a specific end-use properties depending on the glass transition temperature profile (Fig.2b). The monomers used (styrene and butyl acrylate) in the copolymerization process have different reactivity ratios and their polymers have very different glass transition temperature (-54 °C for butyl acrylate and 100 °C for styrene). The key feature of the optimization problem is to determine optimal feed rate and time periods profiles necessary to control polymerization reactions in order to produce particles with a designed morphology and glass transition temperature (Fig.2a). Two objective functions have been selected for the optimization of this process. The first one is to minimize the error between the glass transition temperature and the desired profile. The second objective is to maximize conversion at the end of the process which leads to higher productivity.

$$Min\ \boldsymbol{f} = [f_1, f_2]^T$$
$$f_1 = \frac{1}{t_{fc} - t_0} \int_{t_0}^{t_{fc}} |T_g - T_{g1}|\, dt$$
$$\qquad + \frac{1}{t_{fs} - t_{fc}} \int_{t_{fc}}^{t_{fs}} |T_g - T_{g2}|\, dt$$
$$f_2 = -X(t_f)$$
$$s.t.\quad \dot{\boldsymbol{x}} = \boldsymbol{f}\left(\boldsymbol{x}(t), \boldsymbol{u}(t), \boldsymbol{p}, t \right);\qquad \boldsymbol{x}(t=0) = \boldsymbol{x}_0 \quad (1)$$
$$\frac{1}{t_{fc} - t_0} \int_{t_0}^{t_{fc}} \left(0.9 - X(t) \right)^2 dt \le \epsilon^2$$
$$X(t_0) = 0.9$$
$$\boldsymbol{u}_{inf} \le \boldsymbol{u}(t) \le \boldsymbol{u}_{sup}$$
$$\boldsymbol{x}^T = [V_R, M_1, M_2, M_{T1}, M_{T2}, I, Z, TA, S,$$
$$\qquad N_P, R_1, R_2, \chi_1, \chi_2, \tilde{n}, \lambda_1, \lambda_2, N_m, L_1, L_2]$$
$$\boldsymbol{u}^T = [\Delta t_1, \Delta t_2, \dots, \Delta t_n, Q_1, Q_2, \dots, Q_n]$$

where T_g is the time dependent glass transition temperature, T_{g1} the desired glass transition temperature for the core ($5°C$), T_{g2} the desired glass transition temperature for the shell ($10°C$), t_{fc} and t_{fs} the times necessary to make the core and the shell production respectively, $X(t_f)$ is the overall conversion at the end of the process and $\boldsymbol{u}$ the control vector (feed rates and time periods).

The control variables are time independent parameters and the bounds of these variables are selected according to practical operating conditions.

At the first stage of the process, the primary particles are produced under batch conditions. This stage ends when the overall conversion reaches the value of 0.9. The reactor is then fed with pre-emulsioned monomers and chain transfer agent (CTA). Core stage is designed to be under starving conditions (no droplets are produced and the feed rate is equal to the polymerization rate). Styrene is consumed faster than butyle acrylate due to the difference between there reactivity ratios. As a result, the instantaneous glass temperature will grow to reach the desired value. This stage is operating under a constraint on the overall conversion. The shell stage is conducted without required conditions or constraints. Only the objective to reach the second step of the designed glass temperature

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profile is kept. Feed rates are more important at this stage to allow the growth of the glass temperature by adding more quantities of styrene. The final stage is operating under batch conditions. Since no styrene is added the residual butyl acrylate is consumed leading to lower glass temperatures. The objective at this stage is to maximize the overall conversion which means maximizing productivity and minimizing residual volatile organic compounds (VOC's).

3. DECISION SUPPORT ALGORITHM

The approximation of the Pareto zone obtained with an evolutionary algorithm is a set of compromises (Fig.1). This is an important information for industrialists, but in a second step, we are confronted to a multiple criteria decision problem to classify all nondominated points. Then, the decision maker has to define his preferences based on his knowledge of the process. These expressions allow to propose a decision support system which aggregates all the decision maker's preferences. The decision maker has to express several parameters to define his preferences. He must introduce the weights w_k of each criterion k, depending on the relative importance of the criteria.
The decision support approach used in this work is the multiple attribute utility theory (MAUT). It is a systematic method of identifying and analyzing multiple variables to provide a common basis for arriving at a decision. In the MAUT method, the key element is to derive a multi-attribute utility function for which single utility functions and their weighting factors are necessary. The procedure is as follows (Kim and Song (2009)):

- Setting an objective and establishing the attributes for the goal
- Setting a range of the attributes
- Deriving the single utility functions for each attribute
- Calculating the weighting factors for each attribute
- Deriving the multi-attribute utility function

According to the decision maker preferences the single utility function of the first criterion is chosen as a decreasing function while the second one is an increasing function. The single normalized utility functions used in this work are as follows :

$$g_1(\boldsymbol{u}) = \left(\frac{f_{1\,max} - f_1(\boldsymbol{u})}{f_{1\,max} - f_{1\,min}} \right)^{\alpha_1} \qquad (2)$$

$$g_2(\boldsymbol{u}) = \left(\frac{f_2(\boldsymbol{u}) - f_{2\,min}}{f_{2\,max} - f_{2\,min}} \right)^{\alpha_2} \qquad (3)$$

Where $f_{1\,max}$, $f_{2\,max}$, $f_{1\,min}$, $f_{2\,min}$ are the maximum and minimum values of the first and second objective functions obtained from the Pareto front.
The final multi-attribute utility function is given as a combination of the single utility functions as follows :

$$U(\boldsymbol{u}) = w_1\,g_1(\boldsymbol{u}) + w_2\,g_2(\boldsymbol{u}) \qquad (4)$$

Where w_1, w_2 represent the weighting factors of the utility function ($\sum_{i=1}^{2} w_i = 1$).
The Pareto set elements are ranked according to their multi-attribute utility function value (score) which leads to the best solution (best values of the decision vector) to be implemented.

4. RESULTS AND DISCUSSION

In the preceding sections, the multiobjective problem and the decision aid strategy were presented in details. In the current section, the results obtained from the study are presented. The operating conditions are summarized in Table 3.

Species	Initial charge	Feed charge
Butyl acrylate, (g)	12	48
Styrene, (g)	12	48
Initiator, (g)	1	0
n-C12 mercaptan (CTA), (g)	0.12	0.48
Surfactant (REWOPOL SBFA), (g)	3	12
Water, (g)	114	445
Temperature, ($^{\circ}C$)	70	

Table 3. Composition used for the simulation of the process

The first result of the multiobjective optimization is the Pareto-optimal set of solutions depicting tradeoffs between the competing objectives. This set was generated by using an evolutionary algorithm (EA) with different initial populations. The best results obtained are presented in Fig. 3. The best value of the objective functions taken individually are 4.4 (the error between the designed and the resulting profiles) and -0.948 for the criterion related to the final conversion.
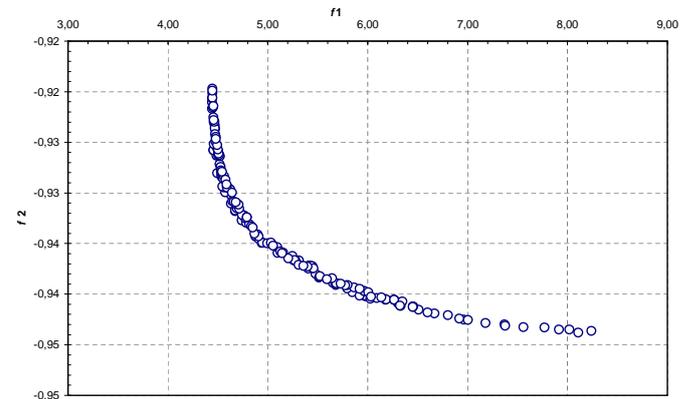
Fig. 3. The Pareto front of the copolymerization multiobjective problem

The decision aid method developed in this work (MAUT) leads to the best solutions according to the decision maker preferences and the utility function used. The weighting factors and parameters of the single utility functions used in this work are given in table 4.

objective function	weight (w_i)	α_i
f_1	0.65	1.5
f_2	0.35	0.5

Table 4. Weights and parameters of the single utility functions

The best profile of the decision variables (time periods and feed rates) obtained are presented in (Fig. 4). It is noteworthy that the feed profile shows clearly the limits of the second and the third stage. The second one (core
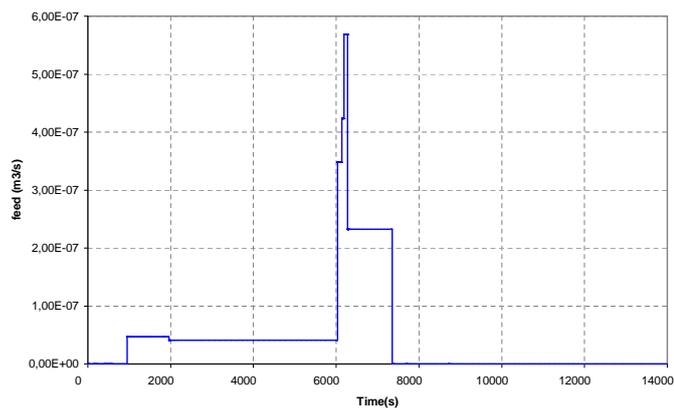
Fig. 4. Feed rate profile of the best solution

Fig. 5. Instantaneous glass temperature profile

Fig. 6. Overall conversion
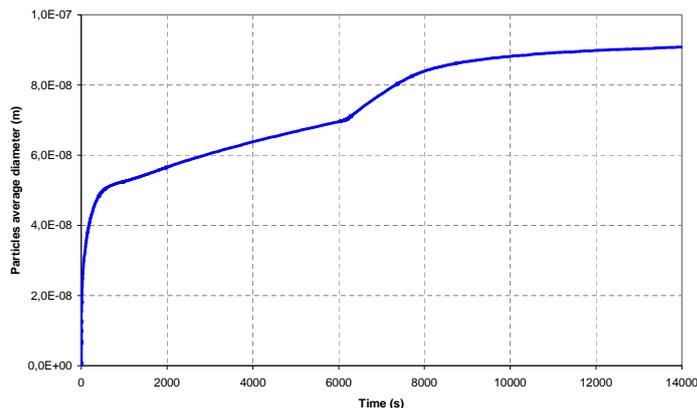
Fig. 7. Average particles diameter
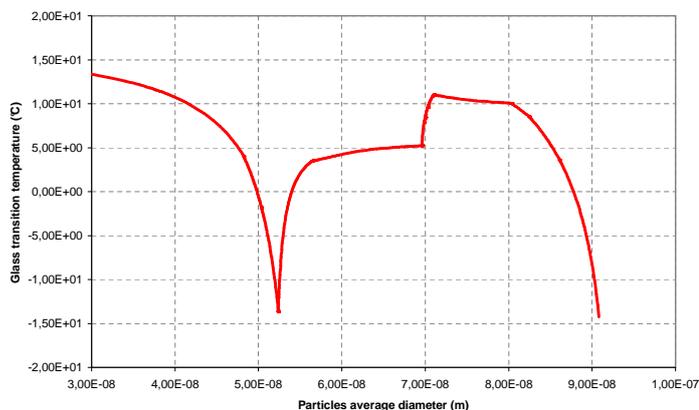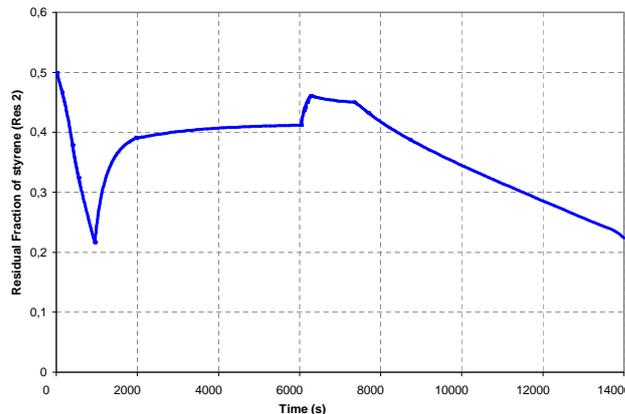
Fig. 8. Styrene residual mass fraction

also noticed at the end the process.

The overall conversion at the stage of the core formation lies in the limit of the constraint (Fig.6). The conversion falls at the shell stage as a result of higher feed rates (no constraints on the conversion are applied). On the other hand, the last stage (batch process) shows that the overall conversion grows to reach the final conversion which is high enough compared to the best solution obtained for the second objective function.

The end-use properties of the final product are related to the average particles diameter. The profile of the average diameter presented in figure 7 shows that the particles diameter grows regularly which means that the number of particles is not changing during the operation. Since no new particles are created during the different stages of the process, the particle size distribution (PSD) is narrow and the morphology of the particles is well controlled and consequently the end-use properties of the product.

The residual fraction of styrene is another relevant information which shows how styrene is consumed during the different stages of the process (Fig.8). Since styrene is more reactive than butyl acrylate, the residual fraction of styrene falls down first with the batch stage and grows up after the start of the feed to reach a constant value related to the designed profile of the glass transition temperature (T_g). This fraction grows once more to reach the second value necessary to the second part of the (T_g). During the

stage) is characterized by low feed rate which corresponds to starving condition where styrene is more consumed leading to the designed glass temperature. Similarly the third stage shows higher feed rate necessary to increase once more the glass temperature.

The implementation results of the best profile are given by Fig. 5-8. First, the glass temperature profile obtained corresponds to the designed profile (T_{g1} = 5 °C and T_{g2} = 10 °C). The first stage (the primary particles formation or seeding) ends with a fall in the glass temperature value. This is quite realistic since butyle acrylate is more consumed when no styrene is added. This phenomenon is

last stage where no monomers are added, the fraction of styrene falls down according to the kinetic of the styrene consumption.

More results related to the number and weight average weights and residual monomers are not presented here for the sake of brevity.

5. CONCLUSIONS

In this work, multiobjective optimization problem has been addressed to determine optimal feed profiles necessary to produce core-shell latex particles with specific end use properties depending on the application (e.g. paints or adhesives). This has been achieved with a designed glass temperature profile and maximum final conversion necessary to maximize production and minimize residual volatile organic compounds (VOCs). The non dominated solutions (Pareto set) were obtained by an evolutionary algorithm developed for this purpose. This set of solution give a wide range of operational options necessary to the improvement of the process. Pareto solutions were ranked by using MAUT strategy. This approach which is based on the decision maker experience and preferences, leads to the unique solution to be implemented. The simulations of the best solution showed a good agreement with the desired profiles.

Finally, the experimental implementations of the best solution are undertaking and the results will be presented in the conference.

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