

Scale-up of adsorptive styrene drying

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Abstract: This work is focused on the analysis of the scale-up process of styrene drying by adsorption onto activated alumina as a necessary step in the manufacture of styrene–butadiene rubber. In a previous work, the mathematical model was developed and the design parameters were estimated from the fitting of the model to experimental results obtained in a laboratory set-up. The obtained model and parameters have been used to simulate the behaviour of a pilot plant that contains up to 56 times more sorbent stating their validity after comparison with experimental results. Finally, the behaviour of an industrial plant presently at work, was also satisfactorily described. The ability of the model to simulate experimental results obtained on three different scales checks its adequacy for process design and optimization.

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Keywords: styrene; drying; adsorption; alumina; scale-up; styrene–butadiene rubber

Notation

a_p	external surface area/volume ($\text{m}^2 \text{m}^{-3}$)
C_i	adsorbate concentration (mg kg^{-1})
$C_{o,i}$	adsorbate initial concentration (mg kg^{-1})
C^*	adsorbate equilibrium concentration (mg kg^{-1})
$C_{i,\text{pore}}$	concentration in pore (mg kg^{-1})
d_p	particle diameter (m)
D_m	molecular diffusivity ($\text{m}^2 \text{s}^{-1}$)
D_p	diffusivity in pores ($\text{m}^2 \text{s}^{-1}$)
E	axial dispersion ($\text{m}^2 \text{s}^{-1}$)
F	flow rate ($\text{m}^3 \text{s}^{-1}$)
k_f	film diffusion coefficient (m s^{-1})
k_m	lumped parameter mass transfer coefficient (m s^{-1})
K_{di}	fraction of interparticle volume species can penetrate
q_i	amount of solute adsorbed onto the solid/amount of solid (kg kg^{-1})
r	radial direction
Re	Reynolds number ($\rho_1 u_s d_p \mu^{-1}$)
R_p	particle radius (m)
Sc	Schmidt number ($\mu D_m \rho_1^{-1}$)
Sh	Sherwood number ($k_f d_p D_m^{-1}$)
t	time (s)
T	temperature ($^{\circ}\text{C}$)
u_i	interstitial fluid velocity (m s^{-1})
u_s	superficial fluid velocity (m s^{-1})
z	axial distance in column (m)
ε_e	external void fraction
ε_p	particle void fraction

μ	viscosity ($\text{kg m}^{-1} \text{s}^{-1}$)
ρ_1	liquid density (kg m^{-3})
ρ_s	structural solid density (kg m^{-3})
ρ_p	particle density (kg m^{-3})

1. INTRODUCTION

Organic compounds are commonly used in many industrial processes. The presence of moisture in those compounds results in poor insulation, corrosion of apparatus and even catalyst poisoning. Therefore, the moisture content of the organic compounds must be reduced to less than several parts per million (ppm) in most processes.¹

Styrene monomer is a raw material in the manufacture of different types of synthetic rubber. Polystyrene is produced on a very large scale. Different copolymers have been designed joining styrene to other molecules such as butadiene, isoprene and acrylonitrile, to obtain materials with a wide range of properties: poly(styrene-co-acrylonitrile) (SAN), acrylonitrile–butadiene–styrene terpolymer (ABS), etc.

Uses for these plastics are extensive and include packaging applications such as disposable tumblers, television cabinets, meat and food trays; rigid foam insulation in various forms used in the construction industry.²

There are several types of polymerization processes. The Spanish company Dynasol Elastómeros, which produces 80 000 t per year of products based on

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styrene, has developed an industrial process of anionic polymerization in solution. This type of reaction is more sensitive to impurities than the free radical system, for instance, and pre-treatment of the monomer is required. *n*-Butyllithium is used as initiator. It is highly susceptible to contamination by polar compounds that cause an excessive and non-desirable consumption of initiator. Among these compounds of special importance are water and 4-*tert* butylcatechol (TBC), a polymerization inhibitor added to avoid homopolymerization during transport and storage. Water appears as an impurity in styrene production and it can also be absorbed from the environment during handling and storage. The value of saturation of water in styrene is 380 mg kg^{-1} at 10°C ,³ although common concentration values are between 100 and 150 mg kg^{-1} .

Nowadays, in many industries that need to purify this type of organic compounds, adsorption on alumina is the preferred technology. However, in most cases this process is empirically controlled. In the case of styrene, it is cooled to $10 \pm 0.3^\circ\text{C}$ (higher temperatures increase the homopolymerization rate) and then it flows through a fixed bed of alumina. The moisture content of the outlet fluid is determined by means of specific probes. When it reaches a non-desirable level, the bed of alumina is changed to a new one.

Activated aluminas cover a wide range of industrial and technical applications. These products are obtained by thermal dehydration of different aluminium hydroxides in the temperature range $250\text{--}800^\circ\text{C}$. Water is driven out upon heating of the hydroxides; a highly porous structure of aluminium oxide having a high surface area remains and activated alumina shows a strong affinity for water. The mechanism of binding of water on the activated alumina surface remains unclear. Present theories favour a mechanism of dissociation of water to H^+ and OH^- ions that become attached to surface sites consisting of an oxide ion on the outermost surface layer and an incompletely co-ordinated aluminium ion in the next lower layer. This exposed cation is located in a 'hole' that is electron deficient and thus acts as a Lewis acid site.⁴

For the proper design of an activated alumina desiccant column, detailed information is required

concerning both the kinetics and equilibrium of adsorption of water. Although studies of adsorption on activated alumina were started in the early 1920s, it was only during the late 1960s that the first studies of adsorption kinetics in the water vapour–alumina system were reported. A substantial body of kinetic data for adsorption on alumina covering a fairly wide range of temperature and concentration has now been accumulated, but some of the results are conflicting or contradictory.⁵ Kinetic studies of water vapour adsorption have been reported by Carter,^{6–8} Marcussen⁹ and Desai *et al.*¹⁰ In the case of drying of liquid mixtures some examples working with toluene,¹¹ xylene,¹² ethanol,¹³ ethylene dichloride,¹⁴ diethyl ether¹⁵ and benzene¹⁶ can be found. Adsorption kinetics for a variety of other sorbates including cyclohexane, hexane, heptane,¹⁷ poly(acrylic acid) and poly(vinyl alcohol)¹⁸ or oxalic, maleic, benzoic and salicylic acids¹⁹ onto alumina have also been reported. Work concerning other adsorbents for drying of organic compounds such as molecular sieves^{11,20–22} or ion-exchange resins¹ has also been reported. Anyway, there are several important reviews in this subject such as those by Basmadjian²³ or Valenzuela and Myers.²⁴

The analysis of adsorption is less reliable than those of other separations. As a result, the basic issue for adsorption is most commonly not *a priori* the design of a process, but instead it is to increase the scale of a small existing experiment. This increase in scale depends more on an analysis of breakthrough curves than on diffusion coefficients, mass transfer coefficients or height of transfer units. The slope of a breakthrough curve depends on an overall mass transfer coefficient, which includes the effects of diffusion and reaction. Understanding this altered performance is the key to improving the efficiency of adsorption.²⁵

The aim of this work is to analyse the validity of the mathematical model and parameters that have been previously obtained working in a laboratory set-up to describe the behaviour of an industrial unit. As a common step the mathematical model will be checked against experimental results obtained in a pilot-plant set-up.

Table 1. Characteristics of the alumina and styrene used in this work

Alumina		Styrene	
Commercial name	Compalox ANV-825	Manufacturer	Repsol SA (Spain)
		Water content	$100\text{--}330 \text{ mg dm}^{-3}$ ^c
Manufacturer	Martinswerk GmbH	TBC content	$10 \pm 0.5 \text{ mg dm}^{-3}$ ^d
		Purity	99.7% minimum ^b
Specific surface area	$219.5 \pm 5 \text{ m}^2 \text{ g}^{-1}$ ^a	Other impurities	Ethyl-benzene (800 mg dm^{-3} max), phenyl acetylene (100 mg dm^{-3} max), aldehydes (75 mg dm^{-3} max), peroxides (15 mg dm^{-3} max), sulfur (30 mg kg^{-1} max)
d_p distribution:	$2.0\text{--}5.0 \text{ mm}$ ^b		
Al_2O_3	91% minimum ^b		

^a Determined by BET specific surface (ASAP 2000 Micromeritics).

^b Data from supplier documentation.

^c Determined by Karl Fisher coulombimetric titration (DL36 Mettler Toledo).

^d Determined by ASTM D 4590–95a⁴⁰ (UV/VIS Lambda 2 Perkin Elmer).

Table 2. Main dimensions of the different adsorption columns

	Laboratory	Pilot plant	Industrial
Length (m)	0.07 0.14 0.28	0.70	3.66
Inner diameter (m)	3.28×10^{-2}	7.79×10^{-2}	1.20
Section (m ²)	8.45×10^{-4}	4.77×10^{-3}	1.13

2. EXPERIMENTAL

The styrene used in this work was manufactured by Repsol, SA in Puertollano and Tarragona (Spain). Its main characteristics are detailed in Table 1. Alumina Compalox AN/V 825 is used as adsorbent and was manufactured by Martinswerk (Germany). Its main characteristics are also detailed in Table 1.

The process that was studied in this work includes recording experimental results in a pilot plant unit as well as the analysis, of the results obtained on an industrial scale. Constructive data are shown in Table 2, whilst Table 3 shows some operational data. Taking into account the operational data on the laboratory scale, two parameters were considered to be the most relevant to determining scale-up: the superficial velocity of the fluid inside the column and the Reynolds number.^{26,27}

All the laboratory experiments were carried out under controlled temperature with jacketed columns to obtain the breakthrough curves at 10 °C. However, at the pilot plant scale it was not possible to control the temperature of the column and therefore experiments were carried out at ambient temperature. This was

Table 3. Operational data for the different adsorption columns

	Laboratory	Pilot plant	Industrial
Flow (m ³ s ⁻¹)	2.83×10^{-7} 5.50×10^{-7} 1.40×10^{-7}	1.67×10^{-6} 3.33×10^{-6}	5.00×10^{-4}
Superficial velocity (m s ⁻¹)	3.35×10^{-4} 6.51×10^{-4} 1.66×10^{-4}	3.49×10^{-4} 6.99×10^{-4}	4.42×10^{-4}
Reynolds number (10 °C)	1.23 2.38 0.608	1.28 2.56	1.62

useful for observing the influence of temperature on the process of adsorption.

Figure 1 shows a flow diagram of the pilot plant set-up. It consists of a tank of 0.2 m³ capacity that includes a level control that permits filling from a main container of styrene as soon as its level decreases. The fluid was pumped by a metering pump (Bran and Luebbe, Germany, E-P31) provided with a frequency variator (Novat, Spain, Series D) and a hydropneumatic pulse dampener (Olaer, France) to eliminate pulsation in the pumped fluid. The system incorporated a basket filter (Bopp and Reuther, Germany, DN10 PN40) placed before the flow meter (Bopp and Reuther, Germany, OI03 Ag41/A4, DN6).

The fixed bed column was made of steel and some of its dimensions are detailed in Table 2. To register the temperature evolution a thermocouple type K (Cole Palmer, USA) and a register (Testo, Spain, 922) were installed.

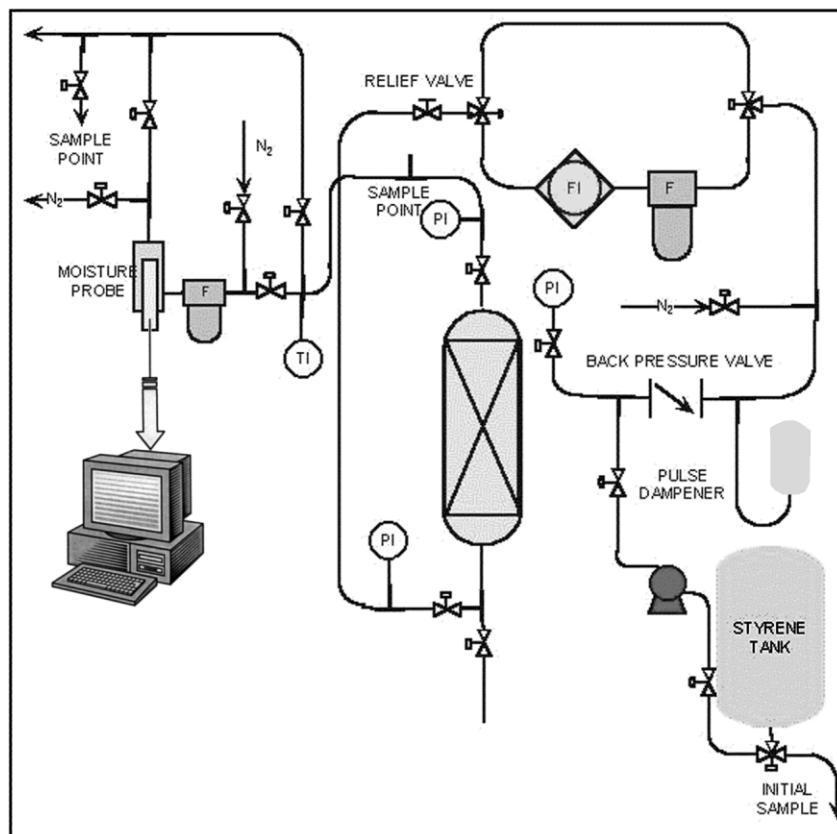


Figure 1. Experimental set-up for adsorption of water from styrene onto alumina in the pilot plant.

To measure the moisture content of the styrene a probe (Panametrics, Ireland, M series) was installed in a sample system that included a removable filter of 50 micron to protect the probe from foreign particles. The heart of the M series probe is Panametrics' proven thin-film, aluminium-oxide moisture sensor, which is capable of measuring dew point/frost point temperatures from -110°C to $+60^{\circ}\text{C}$. The direct measurement of water-vapour pressure was accomplished easily and effectively in both liquids and gases. The sensor consisted of an aluminium strip anodised by a process to provide a porous oxide layer over which a very thin coating of gold was evaporated. The aluminium base and gold layer form the two electrodes of an aluminium-oxide capacitor. Water vapour was rapidly transported on the pore walls of the oxide layer. The number of water molecules adsorbed on the oxide structure determined the conductivity of the pore walls. Each value of pore-wall resistance provided a distinct value of electrical impedance, which in turn was functionally related to the water-vapour pressure. This measuring system was complemented by a computer that was equipped with an acquisition data card.

It is important to take into account the range of validity of the moisture probe. The ambient temperature should be at least 10°C higher than the dew-point temperature. If this condition is not maintained, moisture condensation could occur on the sensor that will lead to reading errors. The accuracy of the probe in the range of working temperature is $\pm 2^{\circ}\text{C}$.²⁸ The concentration of water in styrene in this work was in the range $0\text{--}100\text{ mg kg}^{-1}$ approximately (dew point -45°C to -5°C), so the probe measurements in this range could be considered valid with the accuracy previously mentioned. This type of moisture probe has been used previously by different authors.^{22,29} The results obtained with this probe have been compared to the data obtained using a DL36 Karl Fisher coulometer (Mettler Toledo, Switzerland).

The pilot plant was also provided with a line of cyclohexane that allowed equipment cleaning to avoid styrene polymerization.

Before any experiment started all the system was purged with nitrogen to eliminate residual moisture.

3. MASS TRANSFER MODEL

The differential mass balance to the solute in the packed bed, making the following assumptions: (i) the packing is homogeneous, (ii) there are no radial gradients, (iii) there are no chemical reactions other than adsorption, and (iv) there are no phase changes other than adsorption, can be expressed as

$$\varepsilon_e \frac{\partial C_i}{\partial t} + K_{di}(1 - \varepsilon_e)\varepsilon_p \frac{\partial \bar{C}_{i,\text{pore}}}{\partial t} + \rho_s(1 - \varepsilon_e)(1 - \varepsilon_p) \frac{\partial \bar{q}_i}{\partial t} + \varepsilon_e \frac{\partial(u_i C_i)}{\partial z} - \varepsilon_e(E + D_m) \frac{\partial^2 C_i}{\partial z^2} = 0 \quad (1)$$

where the first three terms represent accumulation of solute in the mobile fluid, in the stagnant fluid in the pores and accumulation of the solute adsorbed onto the solid, respectively; the fourth term is the result of convective flow, and the last term is due to axial dispersion and diffusion.

The pore fluid concentration at the wall $C_{i,\text{pore}}$ can be related to the fluid concentration outside the pores, C_i , by the mass transfer equation across the surface film

$$K_{di}(1 - \varepsilon_e)\varepsilon_p \frac{\partial \bar{C}_{i,\text{pore}}}{\partial t} + \rho_s(1 - \varepsilon_e)(1 - \varepsilon_p) \frac{\partial \bar{q}_i}{\partial t} = k_f a_p [C_i - C_{i,\text{pore}}(R_p)] \quad (2)$$

In this equation k_f is the film mass transfer coefficient in m s^{-1} and a_p is the external surface area per unit particle volume ($\text{m}^2 \text{m}^{-3}$). For spherical particles $a_p = 3/R_p$. The left-hand side of eqn (2) describes the accumulation of solute in the particle, while the right hand side is the transfer rate across the surface film.

The terms $\bar{C}_{i,\text{pore}}$ and \bar{q}_i are the volume average values inside the porous particles.

$$\bar{C}_{i,\text{pore}} = \frac{3}{R_p^3} \int_0^{R_p} C_{i,\text{pore}} r^2 dr \quad (3)$$

$$\bar{q}_{i,\text{pore}} = \frac{3}{R_p^3} \int_0^{R_p} q_i r^2 dr \quad (4)$$

Eqns (1)–(4) must be solved simultaneously with the equilibrium isotherm, initial and boundary conditions. Because this set of equations is difficult to solve, a lumped parameter expression is often assumed; thus

$$\varepsilon_e \frac{\partial C}{\partial t} + u_s \frac{\partial C}{\partial z} + \rho_p(1 - \varepsilon_e) \frac{\partial \bar{q}}{\partial t} = \varepsilon_e E \frac{\partial^2 C}{\partial z^2} \quad (5)$$

$$\rho_p(1 - \varepsilon_e) \frac{\partial \bar{q}}{\partial t} = k_m a_p (C_i - C_i^*) \quad (6)$$

In eqns (5) and (6) the entire particle is treated as having a constant amount of solute adsorbed q , and the stagnant fluid inside the pores is assumed to be in equilibrium with the solid, C_i^* .

The following assumptions have been considered in the present work: isothermal operation, constant liquid velocity through the bed, axially dispersed plug flow, negligible accumulation of adsorbate in the pores, solid granules modelled as spheres, linear combination of mass-transfer resistances: therefore

$$k_m a_p = \frac{1}{\frac{1}{k_f a_p} + \frac{R_p^2}{15\varepsilon_p D_p}} \quad (7)$$

$k_m a_p$ being the overall mass transfer coefficient.

With initial conditions

$$C(z, 0) = C_0$$

$$q(z, 0) = q^*(C_0) \quad (8)$$

Table 4. Experimental planning in pilot plant set-up

Expt	Column			
	length (m)	Flow ($m^3 s^{-1}$)	Alumina (kg)	Time (h)
1	0.70	1.67×10^{-6}	3.550	155
2	0.70	1.67×10^{-6}	3.490	275
3	0.70	3.33×10^{-6}	3.590	100
4	0.70	3.33×10^{-6}	3.557	110

and boundary conditions for $t > 0$,

$$\begin{aligned} \text{at } z = 0, \quad C &= C_0 \\ \text{at } z = L, \quad \partial C / \partial z &= 0 \end{aligned} \quad (9)$$

This system of coupled partial differential equations plus the adsorption isotherm and the accompanying initial and boundary conditions has been solved using gPROMS (general PROcess Modelling System, version 1.6B) developed by Process Systems Enterprise Ltd, UK.

The linear combination of mass-transfer resistances has been previously used by several authors.^{5,11,30} The axial dispersion term was determined from the data proposed by Levenspiel.³¹ To determine the film mass transfer coefficient, k_f , several relationships have been found.^{30,32-35} Finally, the best results have been obtained using the Wilson and Geankoplis correlation eqn (10) which has already been used by many authors.^{11,30,32,35-37}

$$Sh = (1.09/\epsilon_e) \cdot Sc^{(1/3)} \cdot Re^{(1/3)} \quad (10)$$

The value of the diffusivity in the pores was obtained by comparing the experimental breakthrough curves obtained in laboratory scale experiments with the simulated curves. A value of $6.101 \times 10^{-9} m^2 s^{-1}$ was found.³⁸

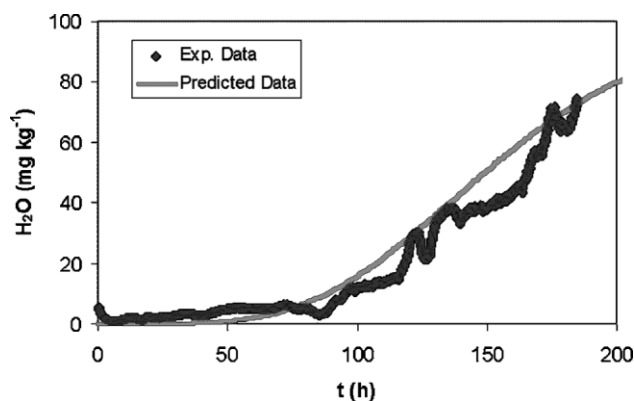


Figure 2. Predicted and experimental results for adsorption of water from styrene onto alumina. Flow rate = $1.67 \times 10^{-6} m^3 s^{-1}$.

4. RESULTS AND DISCUSSION

Table 4 shows the experimental planning developed in the pilot plant. Due to the characteristics of the set-up it was decided to work with the same bed length and different flow rates of styrene.

Before any experiment was done, computer simulations were developed to predict the breakthrough curves. The mathematical tool chosen for this purpose was gPROMS 1.6B. The parameters needed to describe this adsorption system following the model described previously were evaluated taking into account experiments carried out in the laboratory.³⁸

Figures 2 and 3 show the experimental breakthrough curves corresponding to values of two flow rates of styrene together with the curves that were predicted with the mathematical model and design parameters previously obtained.³⁸ Taking into account the fact that the temperature could not be controlled, the simulated data seem to be able to predict the experimental results satisfactorily.

The first experiment is not included in Fig 2 because it was run for several hours daily and was stopped

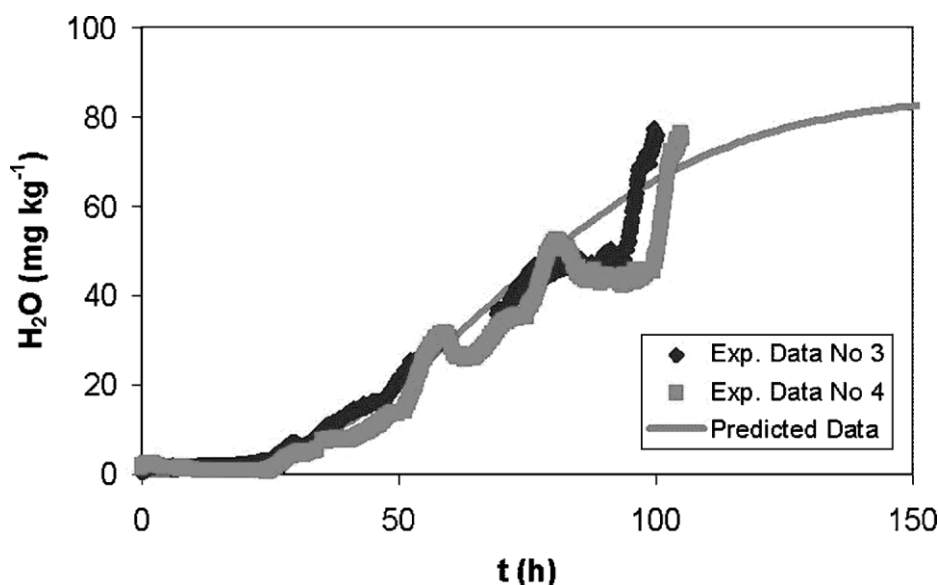


Figure 3. Predicted and experimental results for adsorption of water from styrene onto alumina. Flow rate = $3.33 \times 10^{-6} m^3 s^{-1}$.

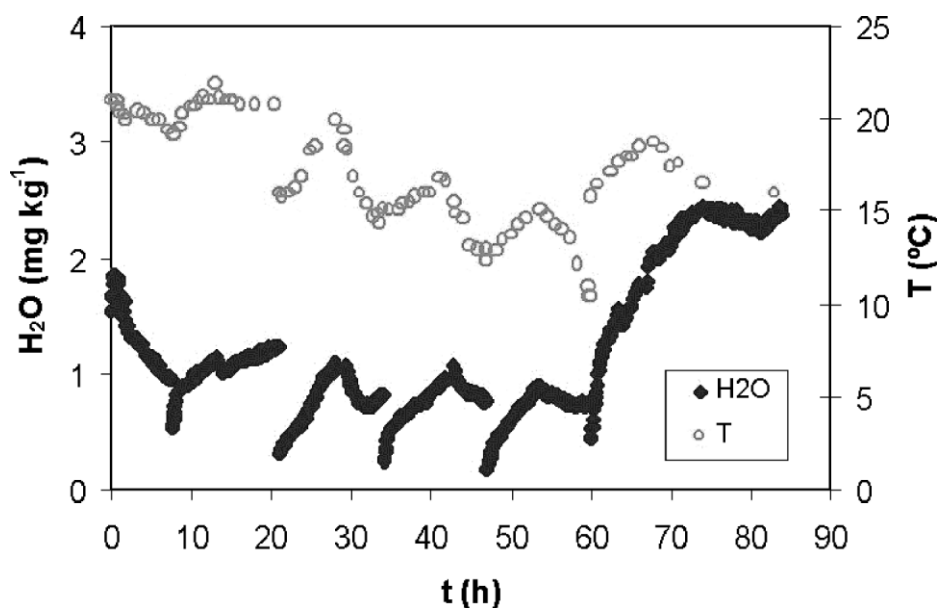


Figure 4. Influence of temperature on the concentration data before breakpoint for experiment 1. Flow rate = $1.67 \times 10^{-6} \text{ m}^3 \text{ s}^{-1}$.

overnight. This seemed to influence the breakthrough curve, so the rest of the experiments were carried out in a continuous mode.

For a constant temperature, before the breakpoint the outlet concentration of water in styrene should be constant and practically zero. Figures 4 and 5 show approximately these parts of the breakthrough curves together with the temperature change. Discontinuities due to the working mode are also appreciated from Fig 4. These graphs allow one to observe, in a qualitative way, the influence of temperature on the adsorption process; when this variable decreases, adsorption of the desired compound increases, which means that the water concentration of the effluent of the column was lower.

Difficulties associated with complete elimination of the residual moisture of the pipes using nitrogen mean

that in the first few minutes of the experiments unexpectedly high moisture contents are detected by the probe, as can be seen in Figs 2 and 5.

Considering the agreement between the experimental results and the values predicted by the model to be acceptable could confirm the validity of the mathematical model and parameters; therefore they were used to predict the breakthrough curves of adsorption of water contained in styrene onto alumina on a larger scale. As was previously mentioned, this is a process that is already working on an industrial scale, but it is just empirically controlled. Tables 2 and 3 show some of the parameters of the industrial plant. Figure 6 gives simulated curves using the model previously described for several values of styrene flow rate in the industrial adsorption column. It is not easy to obtain an experimental breakthrough curve in the industrial

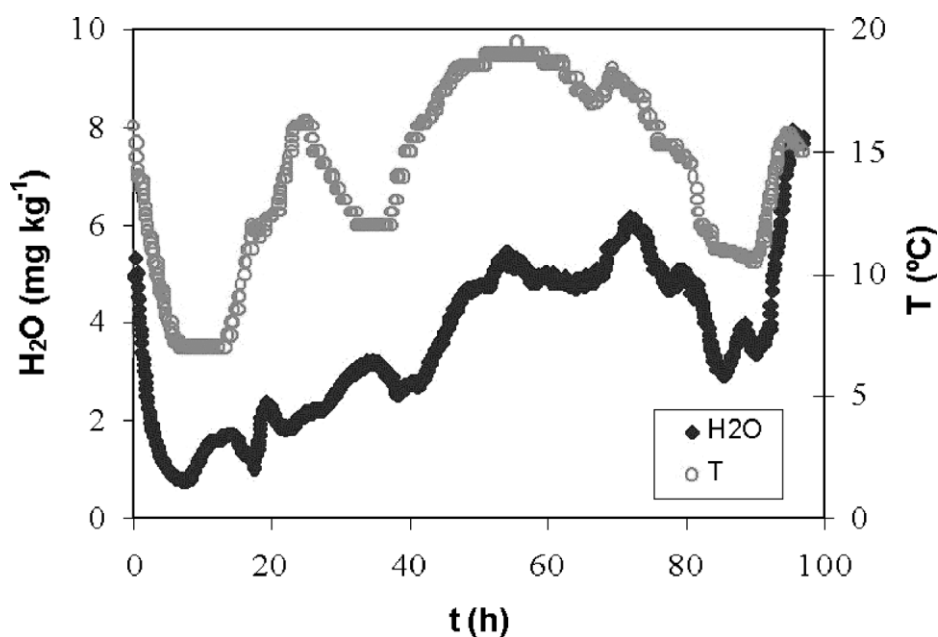


Figure 5. Influence of temperature on the concentration data before breakpoint for experiment 2. Flow rate = $1.67 \times 10^{-6} \text{ m}^3 \text{ s}^{-1}$.

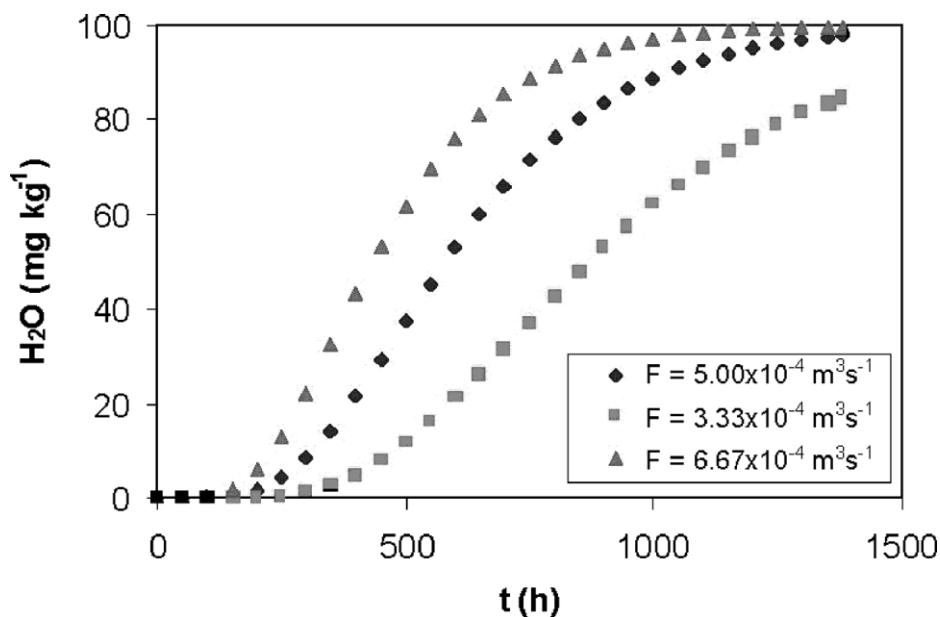


Figure 6. Predicted results for adsorption of water from styrene onto alumina on an industrial scale.

plant because in ordinary work, one line is equipped with three adsorption columns, two of which work in series. The styrene is cooled to 10°C and passed through two columns. Moisture is measured at the outlet of both columns. When the water content reaches a non-desirable level the column is considered useless and therefore the third column starts working, although the previous one is not yet depleted. All available information of the industrial plant has been collected and is shown in Fig 7 together with the curve predicted for the same conditions.³⁹ By comparing both curves it can be concluded that the model describes the adsorption process on industrial scale satisfactorily.

5. CONCLUSIONS

The manufacture of synthetic polymers by anionic

polymerization in solution requires removal of water impurities present in the monomers that would otherwise lead to an excessive consumption of the initiator n-butyllithium.

Considering the manufacture of styrene-butadiene rubber, the adsorption of water from styrene onto alumina was modelled using the data obtained in the laboratory in a previous work.³⁸ The model predictions have been found to be in good agreement with the experimental results of adsorption obtained in a pilot plant set-up, where the ratios of the adsorbent contained in the fixed bed column to that used in the laboratory set-up were approximately 14/1, 28/1 and 56/1, and when the superficial velocity of styrene was kept at similar values. Having checked the validity of the model for predicting the results obtained in a pilot plant it was used to describe the behaviour of the industrial unit where the ratio of adsorbent in the pilot

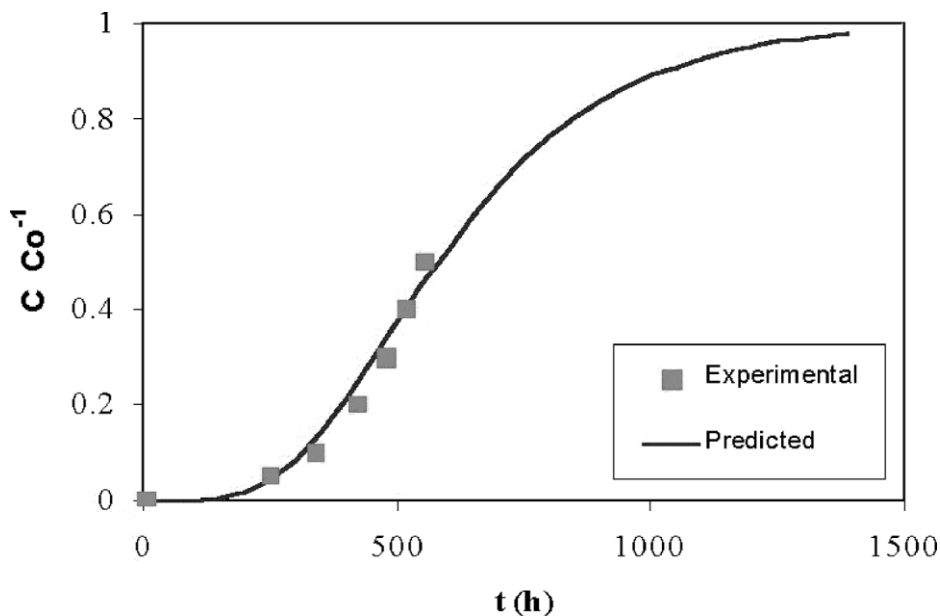


Figure 7. Predicted and experimental data for adsorption of water from styrene onto alumina on an industrial scale ($F=5.00 \times 10^{-4} \text{ m}^3 \text{ s}^{-1}$; $C_0=100 \text{ mg kg}^{-1}$).

plant to that in the industrial plant was 1/1000 and it worked with a similar value of styrene superficial velocity. The simulated results were in good agreement with experimental data collected from the industrial process. Thus, this work confirms the validity of the mathematical model previously developed and provides a useful tool for the prediction of the behaviour and optimization of the industrial process.

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