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Selection of a Purification Process for the Removal and Recycling of Cesium Catalyst after Polyol Production

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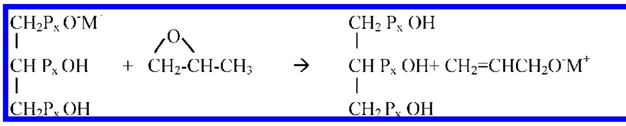
Abstract:

The aim of this study was the development of a crystallization process for the removal of the alkaline catalyst (cesium hydroxide) from crude polyether polyols and its recycling process. Hydrochloric, phosphoric, carbonic, and sulphuric acids were tested to choose the best neutralization agent. Sulphuric acid was chosen because its recycling process seems to be easier than with the other acids assayed. The process involves a first step of neutralization with an aqueous solution of sulphuric acid. After that, the removal of water must be done following an adequate profile of pressure and temperature with time to reach a large particle size that makes possible the filtration of the polyol. The influence of the water/polyol ratio and the *P*−*T* profiles were studied in order to decrease the process time and enlarge the size of the crystals. Once polyol is filtrated, the cake of Cs_2SO_4 was dissolved again to precipitate the sulphate ion with barium hydroxide, thus obtaining a solution of CsOH that can be recycled in the process. It has been confirmed that the recycled cesium hydroxide can be used in a new cycle.

1. Introduction

Polyurethanes (PU) have been used in diverse areas and are one of the most important groups of plastics because of its versatility. The PU market had already reached by the year 2000 world consumption figures of the order of 8.5 million tons. The most important group, the flexible foams, has reached 29% of the production and is widely used in furniture, mattresses, and automotive seats.[1] For the manufacture of high-molecular weight polyurethanes, two kinds of substances are mainly employed: polyisocyanates and polyols. Polyols are also called polyalkylene oxides or polyether polyols. The polyols used in polyurethane manufacture are made from epoxides and have in general an average molecular weight between 200 and 10000, depending on the application. Polyether polyols are usually produced according to the same basic process, namely by reaction, in the presence of a basic alkoxylation catalyst, of an initiator compound having active hydrogen atoms with alkylene oxides, such as ethylene oxide or especially propylene oxide. The typical catalyst used in polyol production, in which the discontinuous batch process is a common practice,[2] is potassium hydroxide. In this process, it is well-known that the unsaturation content increases as a function of molecular weight.[3,4]

Proton abstraction reactions (eq 1) take place especially at higher polymer molecular weight, giving an allylic end-chain adduct as result, which is commonly described as the unsaturation content of a polyether polyol.



$$\begin{array}{l} CH_2P_xO^-M^+ \\ | \\ CH\,P_xOH \\ | \\ CH_2P_xOH \end{array} + \overset{\diagup O \diagdown}{CH_2\text{-}CH\text{-}CH_3} \rightarrow \begin{array}{l} CH_2\,P_x\,OH \\ | \\ CH\,P_x\,OH + CH_2{=}CHCH_2O^-M^+ \\ | \\ CH_2\,P_x\,OH \end{array} \quad (1)$$

In these equations P denotes a propylene oxide unit and M the cation given by the metal hydroxide.

According to the majority of earlier papers[5] we will refer to this reaction as the transfer reaction, which has no effect on the rate of polymerization (i.e., the number of active end chains remains constant) but increases the number of chains (decreased molecular weight), decreases the functionality, and broadens the molecular weight distribution of the product, as the allylic adduct can react with propylene oxide:

$$CH_2{=}CHCH_2O^-M^+ + n\,\overset{\diagup O \diagdown}{CH_2\text{-}CH\text{-}CH_3} \rightarrow CH_2{=}CHCH_2P_n\text{-}O^-M^+ \quad (2)$$

For example, a 6200 g mol^{-1} molecular weight trifunctional poly(propylene oxide) synthesized using KOH as a catalyst (with an ethylene oxide cap) whose functionality should be theoretically 3, will contain between 40 and 45 mol % of non-trifunctional alcohols due to secondary reactions of chain transfer. This fact means that the real functionality will be approximately 2.1.[6]

In order to reduce the unsaturation levels and increase the average functionality, cesium hydroxide can be used as catalyst. The employment of cesium hydroxide as a catalyst for polyol synthesis has been claimed in different patents due to its practical interest.[7−11] Cesium, with a high ionic radius, allows a large separation of the propagating ion pair and favors charge

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separation. As a result of that, there is an increase in reaction rates and a lower level of unsaturation in the final polyether polyol.[6−14] The reduction of unsaturation levels allows to increase the temperature of the process, obtaining an increase in productivity.

The main drawback of cesium hydroxide is its high cost compared to the standard; this basic catalyst is approximately 25 times more expensive than potassium hydroxide. Therefore, it is necessary to find a method to recover and reuse the alkali metal compound in order to provide an economical process.

Regarding the elimination and recovery of the alkaline metal catalysts from polyols, several scientific works can be found in the literature. BASF[14] claimed a method of elimination of the cesium catalyst by means of strong acids. This method included a previous step of a mixture of water and solvent. The solvent was employed in a sufficient excess amount to adjust the differential density between the polyether−solvent solution which is substantially immiscible in water. The amount of the acid was enough to adjust the pH of the mixture to a value not greater than ∼8. The polyether solvent solution is then separated from the water by an electrostatic coalescence method.[15]

Tsutsui et al.[16] proposed a liquid−liquid extraction step, followed by ion-exchange removal of the anion, to obtain an aqueous solution of cesium hydroxide as a result.

Dow Chemical[17] presented a method of recovering cesium hydroxide by extraction with water, that is, phase separation aided with a coalescence treatment. To preserve the polyol from oxidation, the coalescing process was conducted under inert atmosphere. Particulate zirconium dioxide was selected as a coalescing medium.

The objective of this work is the development of a method for the recovery of the cesium catalyst without substantially modifying the standard polyol purification process and using the process plant facilities in the same way that is presently being used conventionally. For the recovery of the catalyst, the crystals of cesium sulphate obtained during the neutralization process are dissolved, and cesium is recovered by chemical treatment. In this process, the solution of cesium sulphate is reacted with a solution of barium hydroxide to precipitate the insoluble barium sulphate salt remaining in the cesium hydroxide solution. After that, the precipitate is separated by filtration, and the cesium hydroxide solution is concentrated and recycled in the process. The recovered catalyst was used again in the synthesis of polyether polyol in order to check its reusability.

2. Experimental Section

2.1. Materials. The experiments were limited to the use of a 6000 MW with and EO tip grade polyol, synthesized as described elsewhere,[19] with cesium hydroxide as catalyst. The alkaline metal content in crude form was ∼4900 ppm (in commercial terms). Its molecular weight distribution was determined by gel permeation chromatography (GPC). A weight-average molecular weight, M_w, of 6000 was obtained.

Sulphuric acid 95−98% (PANREAC PRS-codex grade, Barcelona) was used as neutralization agent. Methanol was used for washing the cesium sulphate cake. Barium hydroxide 97% (PANREAC, Barcelona) was used as the chemical agent to recover the initial catalyst.

2.2. Crystallization Experiments. Batch crystallizations were carried out in a 2-L double jacketed glass reactor, with digital control of stirring rate and temperature. An agitation device equipped with two Rousthon turbines having six vertical blades was used. The vacuum level was controlled by using a Divatronic DT vacuum indicator−controller, acting on a solenoid valve. The experimental setup is shown schematically in Figure 1.

Once the crystallization process was finished, the filtration process was carried out in a thermostat stainless steel cartridge, 10 cm i.d., and having a 2 L capacity at 130 °C and 245 kN/m^2, fitted with a Sartorius glass-fiber prefilter and a cellulose acetate filter with the capability to retain particles greater than 1 μm.

Quality parameters related to the purification process of polyols were also measured. Table 1 shows the standard quality specifications required for these kinds of products.

Cesium content was measured by atomic emission in a Varian Spectra AA 220 FS atomic absorption spectrometer. The polyol samples were diluted with methanol (1:5 by weight) in order to reduce their viscosity.

Water content of polyol was determined by the Karl Fischer method in an automatic TITRINO KF with stirring and suck system TI STAND 703 according to ASTM D 4672-87. Water and iodine are consumed in a 1:1 ratio in the above reaction. The amount of water present in the sample is calculated voltametrically based on the concentration of iodine in the Karl Fisher titrating reagent (i.e., titer) and the amount of Karl Fisher reagent consumed in the titration. Hydranal Composite 5 (Fluka, Germany) and methanol according to Karl Fischer (Panreac, Spain) were used as reagents.

Acidity was measured by automatic titration in a METROHM 721 Net Titrino according to ASTM D 4662-87. To determine the acidity a weighted sample of polyol is dissolved in a 50% mixture of toluene and ethanol and titrated with an ethanolic KOH solution.

Crystal size distribution was determined by using a MASTERSIZER 2000 which is based on the techniqe “low angle laser light scattering” (LALLS).

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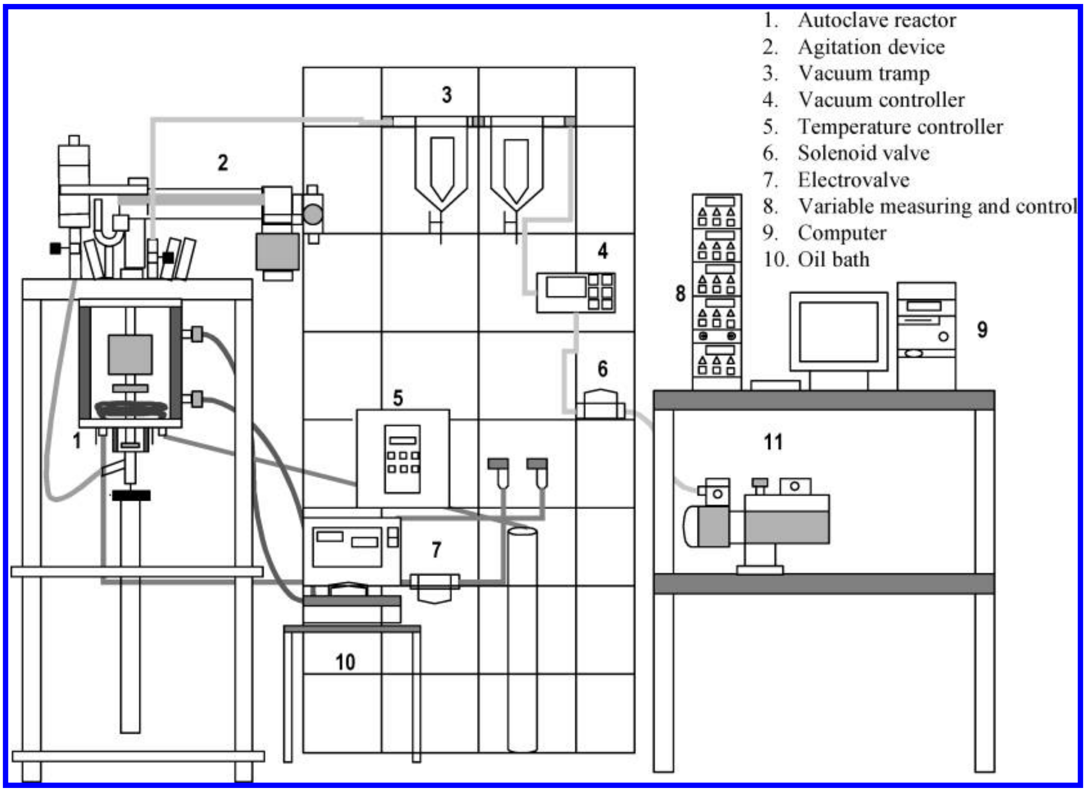

Figure 1. **Schematic of the laboratory-scale crystallizer.**

Table 1. **Quality specifications for flexible glycerol propylene oxide−ethylene oxide-based polyols**

quality parameters	values	test method
sodium and potassium content (ppm, max)	10	ASTM D 4668-87
water content (ppm, max)	800	ASTM D 4672-87
acidity (mg KOH/g polyol, max)	0.1	ASTM D 4662-87

An initial operation scheme was devised on the bases of the literature and our previous knowledge of different industrial polyol processes:

First Step: Monomers Removal. Once the polymerization has finished, a small amount of unreacted monomers remains, contaminating the crude polyether polyol, and they must be removed.

Second Step: Neutralization. If the aim is the formation of crystals, then the second step must be the reaction of the cesium catalyst (polymer bonded) with the neutralization agent solution. An aqueous solution of acid was added in order to neutralize the polyol, forming initially a water−polyol emulsion. The addition of this solution produced a decrease of the bulk temperature.

Third Step: Dehydration−Crystallization. For the elimination of the water in the polyol, a $P-T$ profile protocol of phosphoric acid optimized by A. De Lucas et al.[20] was chosen. It will be called the phosphoric acid curve for simplicity. This curve has been used successfully in processes of neutralization of polyether polyols synthesized with potassium. The experimental conditions are summarized in Table 2.

Fourth Step: Filtration. Once the crystallization process had finished, the filtration process was carried out. The polyol retained in the filter cake was washed out using methanol for analysis purposes. Then a small sample of the filter cake was collected to measure crystal size distribution. The bottleneck of the industrial process is the time employed in the filtration step, which is logically governed by the crystal size distribution. Thus, the main criterion to select the best operating conditions was the time employed for the filtration of the bulk mixture, expressed as the average mass flow G_m (kg/(m^{2}h)). The following criteria were proposed by comparison with the mean times required for the filtration in several industrial polyol processes:

$$\text{Fast filtration: } G_m > 2750 \text{ kg/(m}^2\text{/h)}$$

$$\text{Medium filtration: } 2750 > G_m > 525 \text{ kg/(m}^2\text{/h)}$$

$$\text{Slow filtration: } G_m < 525 \text{ kg/(m}^2\text{/h)}$$

3. Results and discussion

3.1. Preliminary Experiments for Choosing the Neutralization Agent. In order to find the most appropriate neutralization agent, several experiments were carried out using different acids (carbonic acid (in form of CO_2), hydrochloric acid, phosphoric acid, and sulphuric acid (Table 3)) following the experimental procedure described above (Table 2). As can be seen in Table 3, the final cesium content (ppm) of the polyols did not satisfy the standard specification (≤10 ppm). Water specification was satisfied in all cases, whereas the acidity value was nearly the specification.

Although with the use of phosphoric acid the final cesium concentration was 0 ppm, the low solubility of the cesium phosphate salt in water would make too difficult the recovery of cesium. As neutralization agents, hydrochloric and sulphuric acids offered nice results, while the neutralization with CO_2 did not give any remarkable result. Keeping these results in mind and the level

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Table 2. **Summary of experimental conditions for the neutralization curves**

	.			modified profile I			modified profile II		
step	time (min)	pressure (mbar)	temperature (°C)	time (min)	pressure (mbar)	temperature (°C)	time (min)	pressure (mbar)	temperature (°C)
1	0−20	800	110	0−20	800	110	0−20	750	110
2	20−110	750	140	20−50	750	140	20−50	750	140
3	110−170	<20	140	50−110	10	140	50−110	10	140

Table 3. **Preliminary experiments to choose the neutralization agent**

acid	filtrability	cesium concentration (ppm)	acidity (mg KOH/g)	water concentration (ppm)
phosphoric	fast	0	0.1	335
sulphuric	fast	23	0.096	423
hydrochloric	fast	13	0.094	623
carbonic	slow	1223	0.073	427

of difficulty of the possible recovery treatment of the cesium salts, sulphuric acid was chosen because of the treatment with barium hydroxide to obtain a cesium hydroxide solution, that seems to be the easiest way for cesium recovery.

3.2. Influence of the Water/Polyol Ratio. The optimization of the water removal process will be the key factor to obtain large crystals. On the other hand, knowledge of the role that the water plays in the crystallization process is essential to explain the behaviour of the system.[20]

It must be pointed out that the formation of any kind of crystal in polyol media cannot be viewed as a conventional crystal formation process. Obviously, there are not data on the solubility of the different salts on polyols, and also we have to take into account that is not a binary system salt−polyol. Water is playing the key role in the process. At 130 °C water is forming an emulsion with polyol, and the sulphate salts are highly soluble in water. The rate in which water is removed determines absolutely the particle size distribution of the crystal obtained. In this way, the empiricism of the process is great, because it is difficult to know what is happening with the supersaturation of the ternary water−polyol−salts system.

Four experiments, using different amounts of water (10, 5, 2, and 0.5%) in the neutralizing solution, were initially carried out using the same experimental conditions (Table 2). The amount of sulphuric acid used was that stoichiometrically needed to form the cesium sulphate. During these experiments aliquots of the bulk reaction were periodically removed, and the water content and particle size were analyzed (Figure 2).

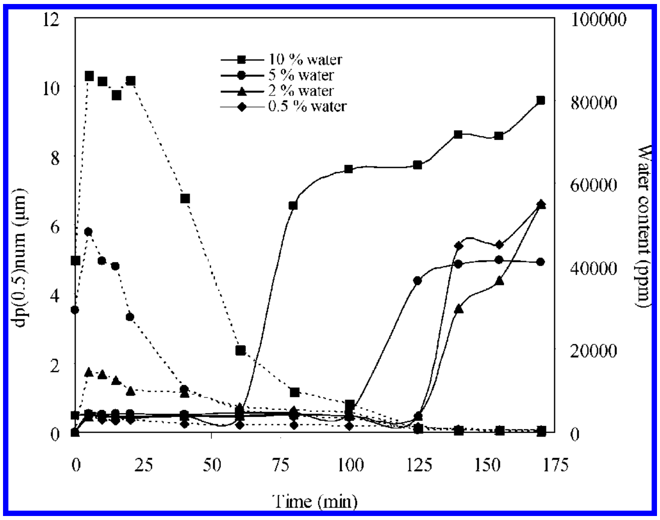

Figure 2. **Variation of crystal size (solid line) and humidity (dash line) with time.**

When the neutralization was made using 10% of water, the final cesium content was 0 ppm. Therefore, the cesium content fell within the specifications. A great increase in the crystal size was observed between 60 and 80 min, that is, in the third step of the neutralization curve (Table 2). In that period (60−80 min) the water content in the polyol decreased from 2% to 1% (Figure 2) coincident with an increase in the size of the particles. It could mean that, in this range of water content of the polyol, the sharp increase of the particle size was happening and it could be possible to reduce the crystallization curve because in the first and second steps of the curve, no crystal formation was observed. In addition, a decrease in the water content could be introduced in the initial formulation because, with only 1−2% of water, the crystal size increase was achieved. The acidity value was measured for the last four samples (125, 140, 155, and 170). This parameter was not within specifications (always higher than 0.1 mg KOH/g).

The initial amount of water used was sequentially reduced to get a reduction of the operation time. When 5% of water was used, the final cesium content was within specifications (9.2 ppm). The appearance of large crystals was produced above 100 min (Figure 2) in the forth and last step of the crystallization curve (high vacuum step). Between 100 and 125 min, the moment in which the crystals started to grow, the water content of the polyol was between 0.3% and 0.05% (Figure 2). As was mentioned above, it would be possible to reduce the crystallization curve time and even the water content for the neutralization. In this case, the acidity could meet the specification (Table 1).

With a water content of 2%, the cesium content (10 ppm) and the acidity value (0.07 mg KOH/g) were within specifications. As in the previous experiment, the point of the quick enlargement of crystal size was reached at 125 min. At this point, the polyol humidity is about 0.1%.

Basically, the system seems to behave in a two-step process:

Step one - comprises the extraction and neutralization of cesium from the polyol phase to the water rich phase of the emulsion
Step two - when the two phases of the emulsion behave like different and separate systems: a polyol phase, without any modification until the filtration process, and an aqueous phase, where the cesium sulphate remains dissolved until reaching the saturation concentration by water evaporation. In any case, the appearance of the water−polyol mixture during the process is like an homogeneous emulsion during the major part of the water-removal process.

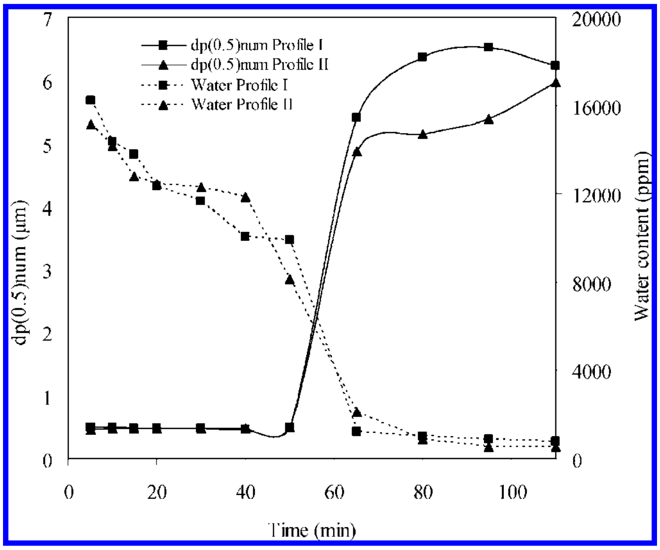

Figure 3. **Evolution of humidity and particle size distribution (2% of water).**

It is really difficult to know the saturation concentration of a complex system like polyol−water−Cs_2SO_4 and what the influence is of each one in the crystal formation. Taking into account the solubility of Cs_2SO_4 in water (179 g Cs_2SO_4/100 g water at 20 °C) and the theoretical amount of the Cs_2SO_4 that has to be formed (4.865 g Cs_2SO_4/kg of polyol), it would be possible to calculate the concentration in order to reach the saturation and subsequent crystal appearance (2.7 g H_2O/kg of polyol). This water content was calculated, assuming no polyol effect. Therefore, the latter experiment was carried out using 0.5% of water.

In this latter case, the final cesium content was 12.7 ppm, and the specification was not met. The result was quite similar to that obtained with a 2% of water (Figure 2), but the crystals formed had a white colour, whereas in the other experiments the crystals had a brown colour.

Except for the experiment using 10% water, in the other ones it was possible to distinguish two periods of time:

- Before 125 min, crystals did not appear, and the water content was very high.
- At 125 min, incipient crystals can be observed at this point with a water content very low. The final cesium concentration was within specifications.

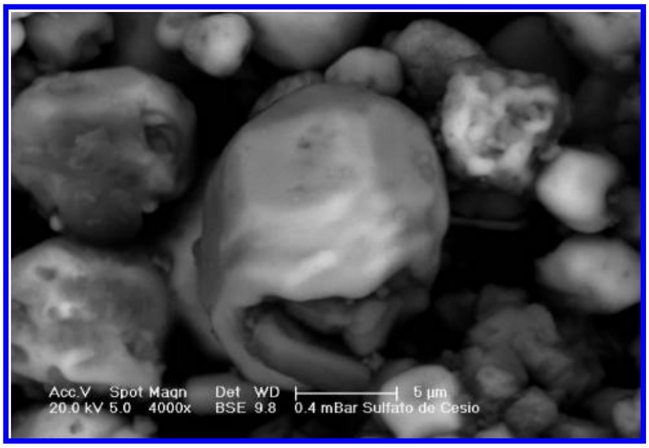

Figure 5. **Scanning emission microscopy of cesium sulphate crystals.**

The curves obtained for different water concentrations, although different in the early stages, were basically coincident from 125 min. Similar results were obtained by De Lucas et al.[19] by using phosphoric acid as the neutralization agent. In this case, the inflection point was nearly 55 min. This fact indicates that, while the water of the dispersed phase was easily removed (under 125 min in our case), the dissolved water was linked to the polyol by hydrogen bonds, making elimination difficult. It seems that the aqueous phase is able to keep the cesium sulphate salt in solution due to its high solubility in water (179 g Cs_2SO_4/100 g H_2O at 20 °C), even taking into account that a very homogeneous emulsion is formed. The nucleation starts when water is almost completely removed and a unique phase of water−polyol−salt appears in the system. The solubility of the salt is really low, and the supersaturation is reached.

No considerable changes in particle size distribution were observed beyond 125 min. Therefore, the crystal growth occurs when the stage of high vacuum starts. In this moment, the water content is approximately 1000 ppm and the time is 125 min.

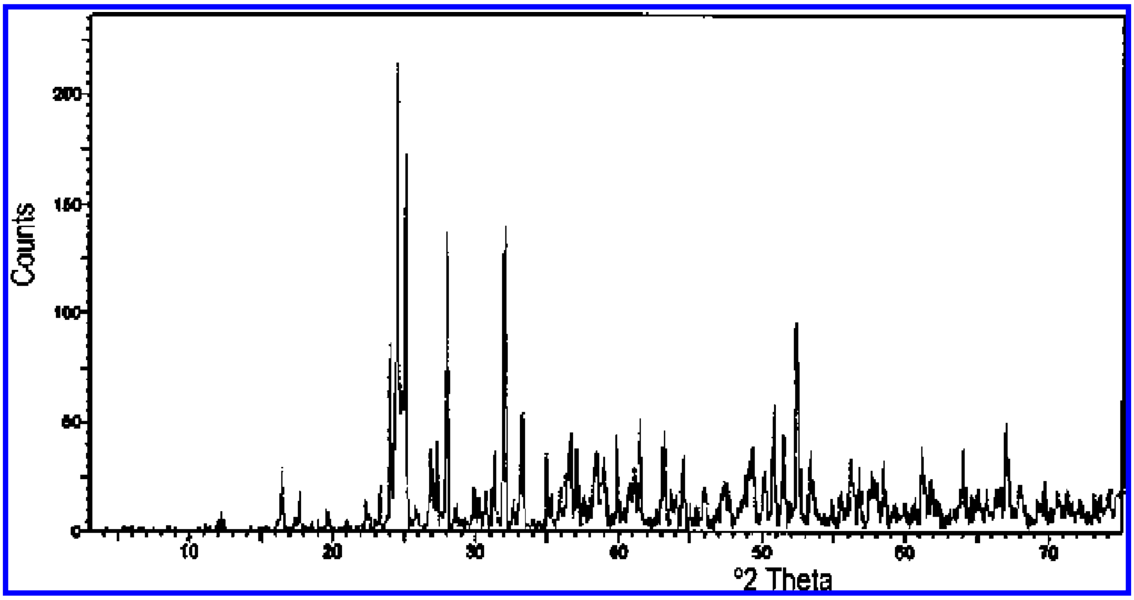

Figure 4. **X-ray diffractogram of cesium sulphate salt.**

This means that the period of time necessary for the dehydration step could be reduced, with the corresponding reduction of the total cycle time. Therefore, in this moment the main goal was to optimize the process conditions in order to reduce the dehydration time. In order to avoid a possible solution of crystals in water, a water content of 2% was chosen for the rest of the experiments.

3.3. Reduction of the Total Cycle Time by Modification of the Process Conditions. As commented above, the crystal growth occurs when the stage of high vacuum starts. This means that the total cycle time can be reduced if the high vacuum stage is forwarded. In order to find out the optimal value for the dehydration time modified $P-T$ profiles were assayed (Table 2). The other operational conditions (stirring rate, water, and acid content) were maintained as in the previous experiments.

Water content in the bulk and particle size distribution during the crystallization process can be observed in Figure 3. At 55 min the first crystals with appropriate size distribution appeared. This fact confirmed that the crystal growth was produced at the beginning of the vacuum stage. Therefore, the period of time necessary for the dehydration step can be reduced to one hour. An average particle diameter of 6 μm was obtained. The quality parameters of the final alkaline metal in polyether polyol satisfied the commercial requirements in the first experiment.

It seems that it a gentle water removal (lower vacuum level) is necessary during the neutralization step to favor the contact between phases.

3.4. Analytical Determination of the Salt Obtained in the Neutralization Process. In order to recover the initial catalyst, it was necessary to confirm that the salt obtained in the neutralization process with sulphuric acid was cesium sulphate. As expected, the diffraction pattern shows sharp peaks, indicating the crystallinity of such a compound. The obtained peak position and subsequently *d*-spacing suggest the orthorhombic crystal structure of cesium sulfate (Figure 4). The morphology of the salt crystals is shown in Figure 5. The SEM micrograph also confirms that the size of the crystals is approximately between 2 and 5 μm.

In order to dissolve the salt for a later treatment, a suitable solvent has to be chosen. In this work, water was used as a solvent because the solubility of cesium sulphate was really acceptable (179 g Cs_2SO_4/100 g H_2O). In addition water is a not toxic solvent, for maximum prevention of hazard in the recovery process.

3.5. Recovery of the Catalyst. The recovery of the catalyst was carried out across a chemical treatment of the salt, cesium sulphate, with barium hydroxide as is shown in the following reaction:

$$Cs_2SO_4 + Ba(OH)_2 \rightarrow BaSO_4 + 2CsOH$$

First, cesium sulphate was dissolved in water. After the complete dissolution, barium hydroxide was added in stoichiometrical amount in order to prevent impurities of ions in the CsOH solution. Finally, the precipitate was separated by filtration. The precipitate of $BaSO_4$ was further analyzed, and less than 1 ppm of Cs was found present in the barium sulphate crystals.

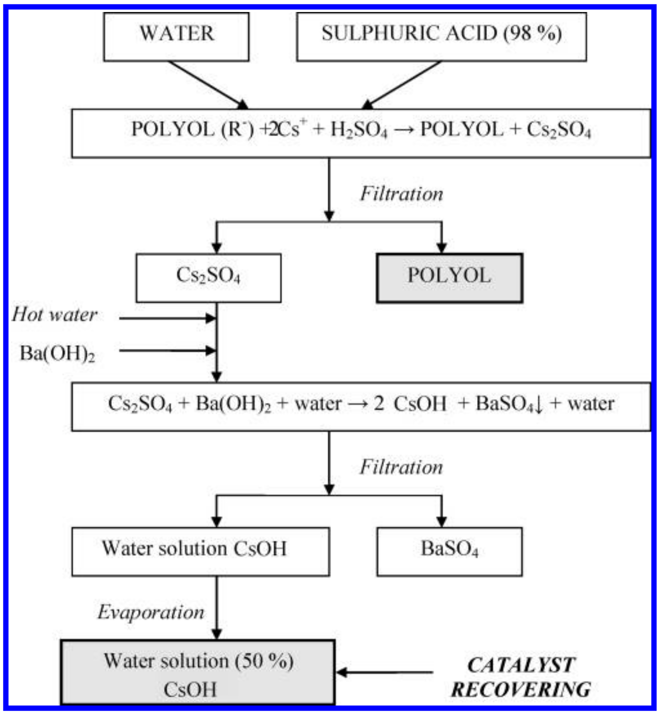

Figure 6. **Scheme for catalyst recovery.**

Second, the solution of cesium hydroxide obtained by the as-mentioned chemical treatment was analyzed. It was found that the solution was practically free of barium impurities (3−4 ppm). The solution could be concentrated up to 50% for its later use in the synthesis of polyols. Therefore, the feasibility of the proposed method to obtain a closed life cycle without catalyst losses is demonstrated and presented in Figure 6.

4. Conclusions

A complete study concerning the neutralization and recovery of cesium catalyst in a polyol production process has been carried out. Several neutralization agents were tested in order to neutralize the cesium hydroxide catalyst used in the polyol production in the laboratory. As the neutralization agent, hydrochloric and sulphuric acids obtained good results. Sulphuric acid was chosen due to the possibility of recovering the catalyst by chemical treatment of the salt with barium hydroxide. Independently of the amount of water used for neutralization, the crystal growth occurs when the stage of high vacuum starts. It has been possible to reduce the period of time necessary for the dehydration step to one hour. Finally, the catalyst could be recovered by a chemical treatment of the cesium sulphate salt with barium hydroxide, and a new polyol could be produced with the recovered catalyst.

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