EVALUATION OF THE REHYDRATION PROCESS OF AN ION EXCHANGE RESIN MADE FROM SULFONATION AND CROSSLINKING POLYMER MATRIX OF RECYCLED EXPANDED POLYSTYRENE

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ABSTRACT

Fundamental research consists in the evaluation of the rehydration process of an ion exchange resin made from sulfonation of polystyrene, used in packaging and packing, in order to determine whether a significant decrease in ion exchange capacity exists. A primary evaluation to the resin, was performed, determining the parameters established in ASTM D 2187-94 (2004), to characterize it, providing a basis for comparison between the initial resin and the rehydrated. Also, these properties were compared with the parameters established for, PUROFINE PFC-100 commercial resin, determining that the resin made exhibit similar features as the comercial one. It was performed, three processes of dehydration and rehydration consecutive, evaluating within each process, the total ion exchange capacity of the resin and establishing the percent decrease thereof. Finally, it was determined that for all dehydrations made, the percent decrease in exchange properties was less than one percent. Recommended as the optimum maximum, three dehydrations by resin.

Keywords: *recycled polystyrene, hydration, total ion exchange capacity, percent decrease.*

RESUMEN

La investigación consistió en la evaluación del proceso de rehidratación de una resina de intercambio iónico, elaborada a partir de la sulfonación de poliestireno expandido, utilizado en empaques y embalaje, para determinar si existe una disminución significativa en su capacidad de intercambio iónico. Se realizó una evaluación primaria a la resina, determinando los parámetros establecidos en la Norma ASTM D 2187 – 94 (2004), para caracterizarla, creando un parámetro de comparación entre la resina inicial y la rehidratada. Asimismo, estas propiedades se compararon con los parámetros establecidos para la resina comercial, Purofine PFC-100, determinando que la resina elaborada presenta características similares a esta. Fueron realizados tres procesos de deshidratación y rehidratación consecutivos, evaluando entre cada proceso la capacidad total de intercambio iónico y estableciendo el porcentaje de disminución de la misma. Finalmente, se determinó que para todas las deshidrataciones el porcentaje de disminución de las propiedades de intercambio fue menor al uno por ciento. Recomendando como límite máximo óptimo, tres deshidrataciones por resina.

Palabras clave: *intercambio iónico, sulfonación, poliestireno, hidratación, capacidad total de intercambio iónico, porcentaje de disminución.*

INTRODUCTION

The ion exchange resins are three-dimensional hydrocarbon chains, to which have been added ionizable groups, through processes of sulfonation, amination or analogous; resulting from them cationic and anionic exchange resins.

By subjecting the expanded polystyrene waste in a process of sulfonation and crosslinking the polymer matrix; it permits sulfonic and sulfone groups link the styrene monomer unit groups. This provides a hydrophilic character and the ion exchange capacity to the material. Thus obtaining an alternative to recycling polystyrene, generating a cation exchanger with low energy consumption and low cost.

In this process, it is obtained as a result, a resin, composed mostly of water. This presents a difficulty in handling the resin. So, it is considered important, the evaluation process of dehydration and rehydration of it. And the regeneration of its main properties, determining whether there is significant loss in its total ion exchange capacity.

ASTM D2187 - 94 (2004) refers to evaluation methods covering the determination of physical and chemical properties of ion exchange resins used in water treatment properties. These methods are intended to the evaluation of new and reclaimed material; making it possible to determine the decrease in exchange capacity of the resin due to hydration and rehydration process.

METHODOLOGY

The project includes five stages: pre-treatment and recycling of expanded polystyrene, sulfonation of the polymer matrix and preparation of the sodium salt of the ion exchange resin, primary evaluation of the resin, dehydration and rehydration, and secondary evaluation of the ion exchanger.

Pre-treatment and recycling of expanded polystyrene

It was applied a pre-treatment process to the material used to produce the resin, consisting of expanded polystyrene degassing, to reduce the volume of the spheres and optimize the space available for the sulfonation.

Sulfonation of the polymer matrix and preparation of the sodium salt of the ion exchange resin

It was used as sulfonating agent a mixture of fuming sulfuric acid, oleum, as a ten percent and concentrated sulfuric acid as the ninety percent and applying a sulfonation time equal to two hours; following the recommendations given in the research *Development of an ion exchange resin from the sulfonation and crosslinking expanded polystyrene recycling for the separation of metal ions in solution.* After sulfonation, the sulfonated polystyrene was neutralized in their acid form, using a sodium hydroxide solution, to bring the hydrogen potential of the mixture to a value greater than ten. Obtaining the sodium salt of the resin. Washings were done with demineralized water to remove excess acid that may be free therein.

Dehydration and rehydration

For the dehydration process a convection oven was used, at a temperature of 104 °C for 18 hours. It was made a mass reconstitution of the resin, adding demineralized water to the dried sample.

Evaluation of the resin

It was performed an evaluation of the properties of the resin, using as quantitative technique, the parameters set in ASTM D 2187-94 (2004). It was determined the backwash density, high water retention, capacity of salt splitting and total capacity of the ion exchange resin and the regeneration capacity of these properties after rehydration.

PRESENTATION OF RESULTS

In the process of sulfonation and crosslinking of the polymer matrix of expanded polystyrene, it is obtained as a result, a resin composed mostly by water. This presents a difficulty in handling the resin and an increase in cost thereof. Which decreases its economic feasibility and marketability. For this reason, it is considered important, the evaluation of the process of dehydration and rehydration of the resin.

Ion exchange capacity of the initial resin.

Chart I. **Total ion exchange capacity of the initial resin obtained by sulfonation of polystyrene**

Ion Exchange Capacity	
Per mass of wet resin, (meq/g)	9,716
Per mass of dry resin, (meq/g)	149,253
Per milliliter of settled and backwashed material, (meq/mL)	10,002

Source: Compiled based on results.

Initial characterization of the resin according to ASTM D 2187-94 (2004)

Chart II. **Determination of characteristics of the resin obtained by sulfonation of polystyrene**

Backwash density (g/mL)	1,03
Maximum percent retention of water (%)	93,49
Salt-splitting capacity per mass of wet resin (meq/g)	0,137
Salt-splitting capacity per mass of dry resin (meq/g)	2,101
Salt-splitting capacity per milliliter of settled and backwashed material (meq/mL)	0,141

Source: Compiled based on results.

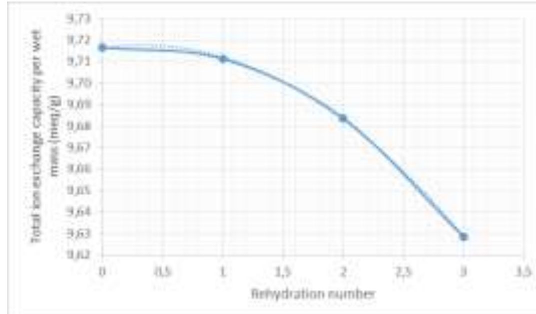
The evaluation of the resin was oriented in the determination of the total ion exchange capacity, and the effect produced by the process of dehydration and rehydration. To meet this goal, the total ion exchange capacity of the starting resin in milliequivalents per gram of wet resin, per gram of dry resin and per milliliter of settled and backwashed material were determined; these results are shown in chart I.

In order to establish a basis for comparison of the rehydrated resin to the initial characterization, it was conducted the evaluation, done with the parameters established as optimal in the previous investigation. Thus backwash density, high water retention capacity and the ability of a neutral salt splitting of the resin were determined. The results are shown in chart II. The assessment was made based on the methods described in ASTM D 2187-94 (2004), "Standard Methods of evaluation of the physical and chemical properties of ion exchange resin particle properties."

Performance of the total ion exchange capacity of the rehydrated resin regarding to the initial

In figures 1, 2 and 3, the performance of the total ion exchange capacity per wet gram, dry gram and per milliliter of settled and backwashed of rehydrated resin regard to the initial is patterned. This model fits a second order polynomial behavior, which has a maximum in the initial resin and a decreasing trend to a minimum.

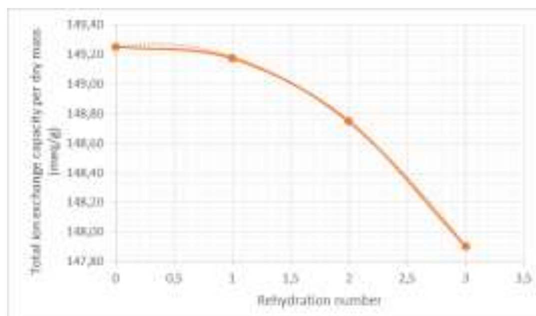
Figure 1. **Performance of the total ion exchange capacity per wet mass of the rehydrated resin regarding to the initial**



Color	Mathematical model	Correlation coefficient	Validity interval
(blue)	$C_w = -0{,}0125R^2 + 0{,}0083R + 9{,}7161$	0,9997	[0,3] R

Source: Compiled based on results, with Microsoft Excel 2013.

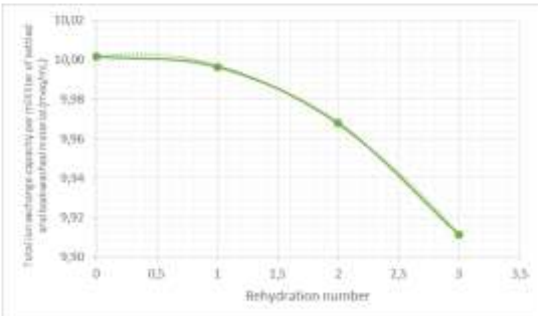
Figure 2. **Performance of the total ion exchange capacity per dry mass of the rehydrated resin regarding to the initial**



Color	Mathematical model	Correlation coefficient	Validity interval
(orange)	$C_d = -0{,}192R^2 + 0{,}1281R + 149{,}25$	0,9997	[0,3] R

Source: Compiled based on results, with Microsoft Excel 2013.

Figure 3. **Performance of the total ion exchange capacity per milliliter of settled and backwashed material of the rehydrated resin regarding to the initial**



Color	Mathematical model	Correlation coefficient	Validity interval
	$C_b = -0{,}129R^2 + 0{,}0086R + 10{,}002$	0,9997	[0,3] R

Source: Compiled based on results, with Microsoft Excel 2013.

Decreased in total ion exchange capacity of the resin in order to rehydration process

Chart III. **Percent decreased in total ion exchange capacity of the resin in order to rehydration process**

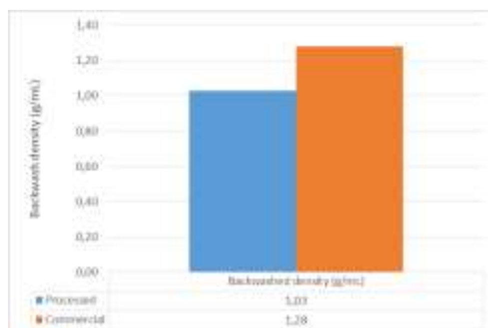
Rehydration number	Percent decreased
1	0,05%
2	0,34%
3	0,91%

Source: Compiled based on results.

The percentage decrease of the total ion exchange capacity in the resin due to each rehydration was evaluated. This rate of increase is, in all cases, less than one percent, this shows that the hypothesis proposed in the investigation, exposing that it is possible to conduct the dehydration and rehydration of a resin obtained from the sulfonation of polystyrene foam of recycled origin, without any significant loss in ion exchange capacity, is affirmative. Likewise, the correlational hypothesis, which states that the total ion exchange capacity of the resin decreases by ten percent in each regeneration, has been proven.

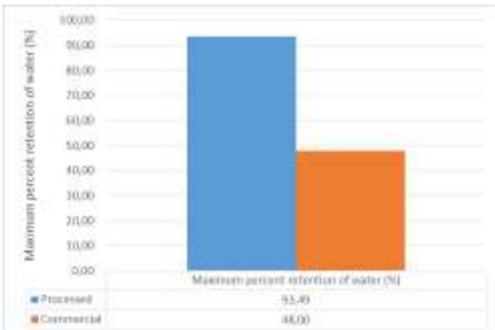
Comparison of characteristics of the processed resin to PUROFINE PFC-100 commercial resin

Figure 4. **Comparison of Backwash density of the resin made of polystyrene to Purofine PFC-100 commercial resin**



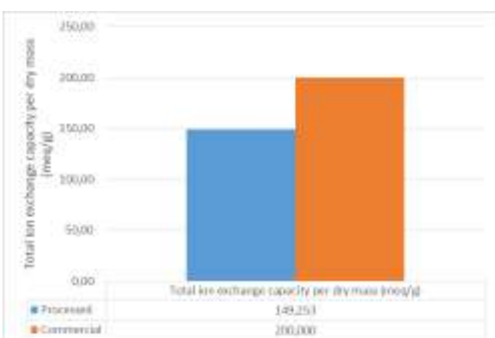
Source: Compiled based on results, with Microsoft Excel 2013.

Figure 5. **Comparación del porcentaje de retención máxima de agua de la resina de elaborada a partir de poliestireno expandido de origen reciclado, respecto a la resina comercial Purofine PFC-100**



Source: Compiled based on results, with Microsoft Excel 2013.

Figure 6. **Comparación de la capacidad total de intercambio iónico de la resina de elaborada a partir de poliestireno expandido de origen reciclado, respecto a la resina comercial Purofine PFC-100**



Source: Compiled based on results, with Microsoft Excel 2013.

The determined properties were compared with commercial sulfonated polystyrene resin Purofine PFC-100. The density of backwashing, as the total ion exchange capacity, figure 4 and 6, are lower in the processed resin than in the commercial, this is because clots present in the gel, reduce the density backwash as increases the amount of water retained in the polymer net. The ion exchange capacity is lowered due to decreased surface area of contact of the resin with the ion exchange medium.

Backwash density determines the density of the material in its maximum water retention, this value is greater than the average density of water at the conditions under which the experiment was performed; it promotes circulation of the resinous material in the backwash, while improving the regeneration of the resin. It is seen in figure 5 that the maximum water retention, unlike the other properties, to the resin is made higher than for the commercial. If the resin has a greater number of sulfonic groups increases solubility of the material and decreases the percentage of high water retention. Since the resin is prepared on a laboratory scale, the sulfonation is weaker than that used for commercial resin; generating a lower proportion of sulphonic groups present in the polymer network.

CONCLUSIONS

1. The total ion exchange capacity per wet mass of the initial resin obtained by the sulfonation of polystyrene is 9,716 meq/g.

2. The total ion exchange capacity per dry mass of the initial resin is 149,253 meq/g.

3. The total ion exchange capacity per milliliter of settled and backwashed material of the initial resin is 10,002 meq/g.

4. The values obtained for the total ion exchange capacity of the resin made

from recycled polystyrene sulfonation origin, correspond to the average value established in ASTM D 2187-94 (2004).

5. The total ion exchange capacity of the resin rehydrated regarding the initial, performs a second order polynomial model, having a maximum in the initial resin and a downward trend to a minimum.

6. The percent decrease in total ion exchange capacity of the resin due to the dehydration and rehydration process is less than one percent, for all trials.

7. The backwash density is lower for the ion exchange resin made from sulfonation of polystyrene recycled origin, than commercial sulfonated polystyrene resin Purofine PFC-100.

8. The ion exchanger processed has developed smaller proportion of sulfonic groups than the commercial, hence its ion exchange capacity is less.

9. The resin produced has a higher maximum percent retention of water compared to the commercial resin evaluated.

10. The backwash average density of the prepared resin is 1,03 g/mL.

11. The average resin made allows maximum water holding 93,49 %.

12. The salt splitting capacity of the initial resin obtained by the sulfonation of polystyrene per wet mass is 0,137 meq/g, per dry mass of the initial resin is 2,101 meq/g, and per milliliter of settled and backwashed material is 0,141 meq/g.

RECOMMENDATIONS

1. Set limit regeneration of ion exchange resin in three dehydrations.

2. Check the temperature of the dehydration process of the resin does not exceed 104 °C.

3. Avoid splitting spherules when performing the backwash process, using a column that holds the height corresponding to the volume of resin to evaluate.

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