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Open-Cell Polyurethane Foam Sponge as a "Solvent Extractor" for Gallium and Iron

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Abstract

Open-cell polyurethane foam has been shown to be effective in separating gallium and iron from aluminum in acid chloride solutions. Evidence is presented which indicates that the solid organic polymer acts as a "solvent extractor" for complex acid chlorides of gallium and iron.

INTRODUCTION

The use of solvent extraction in industrial processes and analytical separations is well known. The work by Bowen (*1*), Braun (*2*), and Gesser (*3*) has shown that polyurethane foam can be used to extract and/or complex inorganic ions and dissolved organic substances from water. The subject has recently been reviewed by Braun (*2*).

The solvent extraction of gallium chloride from acid solutions has been studied for the past 50 years (*4–6*) and is the basis of several analytical separation methods for gallium (*7*, *8*).

In the course of some work on the extraction and recovery of gallium from tantalum tailing, we have found that polyurethane foam with a polyether backbone can be used for the extraction and recovery of gallium from acid chloride solutions.

EXPERIMENTAL

Commercial sheets of open-cell polyurethane foam sponge (OCPUFS) were used. Cylinder plugs of 1¼ in. diameter and 1¼ in. long were cut from the sheets and inserted into 1¼ in. i.d. glass chromatographic columns fitted with Teflon stoppers.

Standard gallium solutions were prepared by dissolving pure metal in concentrated hydrochloric acid, evaporating the excess acid, and diluting the solution to an appropriate volume. Analysis of gallium was performed by atomic absorption spectrometry (*9*).

In order to avoid channeling when solutions were passed through the form plugs, all the trapped air was removed from the plug. This was accomplished by applying a soft vacuum to the solution-filled column until no more air bubbles were released from the foam plug. The solution was then allowed to flow through the plug at the desired flow rate.

RESULTS AND DISCUSSION

The results of the effect of the hydrochloric acid strength on the extraction efficiency of two types of polyurethane foam are shown in Fig. 1 where 50 ml of a solution containing 50 ppm Ga was passed through the

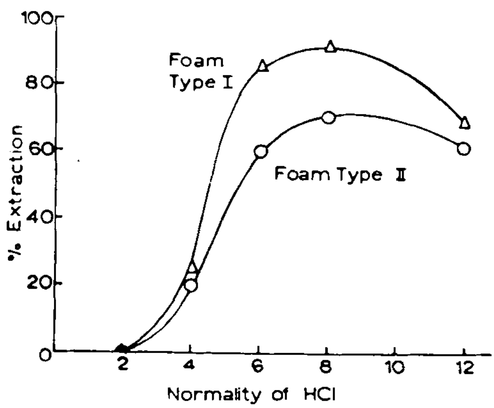

FIG. 1. Effect of HCl normality on the extraction efficiency of gallium from solution by two types of foam. Volume of solution was 50 ml of 50 ppm Ga. This was passed through a foam plug at a flow rate of 64 ml/min at room temperature.

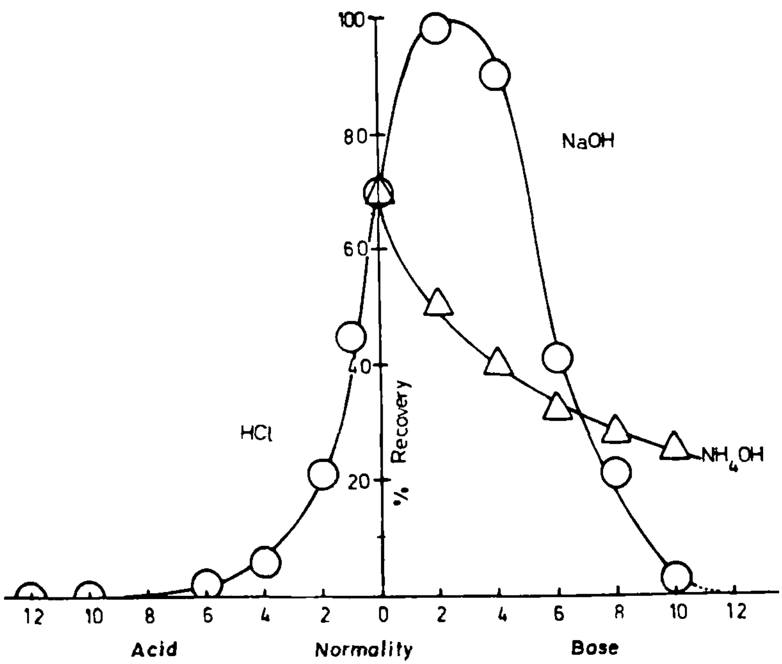

FIG. 2. Effect of acid and base concentrations on the recovery efficiency of gallium from a foam plug. The gallium was absorbed into the foam by 3 passes of 100 ml solution 7.5 *N* HCl containing 50 ppm Ga. Volume of solution was 50 ml of acid or base solution passed through the foam at a flow rate of 64 ml/min at room temperature.

foam plugs at a flow rate of 64 ml/min at room temperatures. Similar results showing a maximum were obtained (*4*, *5*) with ether extraction. The fall off at high concentration of acid, also found in ferric chloride (*10*), has been attributed to a salting-out effect.

Ground-up closed pore rigid polyurethane foam, though less efficient than open-cell polyurethane foam sponge, showed similar extraction characteristics whereas open-cell silicone rubber sponge shows no measurable extraction of gallium under comparable conditions.

The recovery of gallium from the foam was established by absorbing gallium on a foam plug by 3 passes of 100 ml of 50 ppm of gallium in 7.5 *N* HCl. The gallium was then removed by passing 50 ml of various solutions through the foam plug at a flow rate of about 64 ml/min. The results of analysis are shown in Fig. 2 for acidic and basic wash solutions and indi-

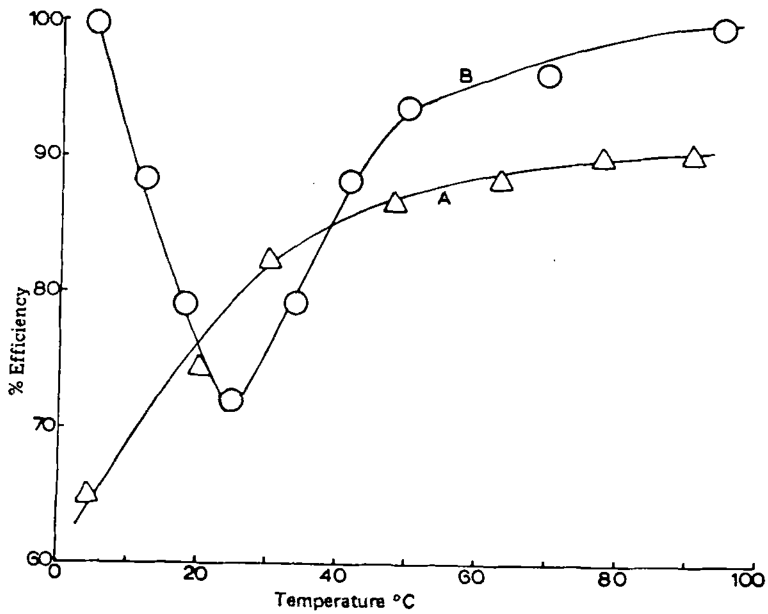

FIG. 3. Effect of temperature on (A) extraction and (B) recovery efficiency of gallium. A: Fifty milliliters of 50 ppm Ga in 7.5 *N* HCl passed through one foam plug at a flow rate of 64 ml/min. B: Gallium was absorbed on the foam with 3 passes of 100 ml of 50 ppm Ga in 7.5 *N*HCl. The recovery was effected by passing 50 ml of water through the foam at a flow rate of 64 ml/min at various temperatures.

cates that water or weakly basic solutions are very effective in removing gallium from OCPUFS.

The efficiency of OCPUFS for the extraction of gallium from the acid solution as well as the recovery of the gallium off the OCPUFS by water is temperature dependent as well as flow-rate dependent. The results of analysis showing the effect of temperature is shown in Fig. 3 and the effect of flow rate is shown in Fig. 4. At lower flow rates of water the recovery efficiency of gallium from OCPUFS increases from 75% to over 95% at 25°C (Fig. 3). The minimum of Curve B in Fig. 3 is probably due to a kinetic effect superimposed on a temperature variation in the thermodynamic equilibrium distribution constant. Thus larger contact time between solution and foam results in efficiency increasing up to 100% for either

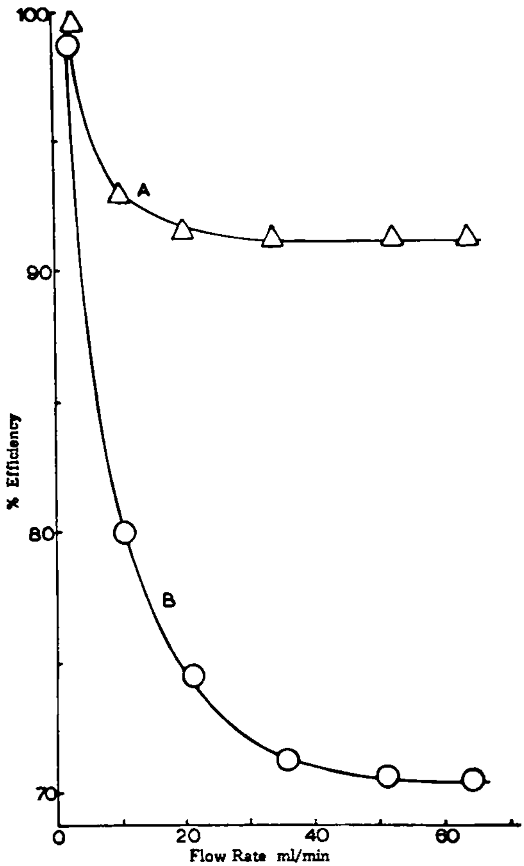

FIG. 4. Effect of flow rate on the (A) extraction and (B) recovery efficiency of gallium. A: Fifty milliliters of 50 ppm Ga in 7.5 N HCl passed through one foam plug at room temperature. B: Gallium was absorbed on the foam with 3 passes of 100 ml of 50 ppm Ga in 7.5 N HCl. The recovery was effected by passing 50 ml of water through the foam at room temperatures and various flow rates.

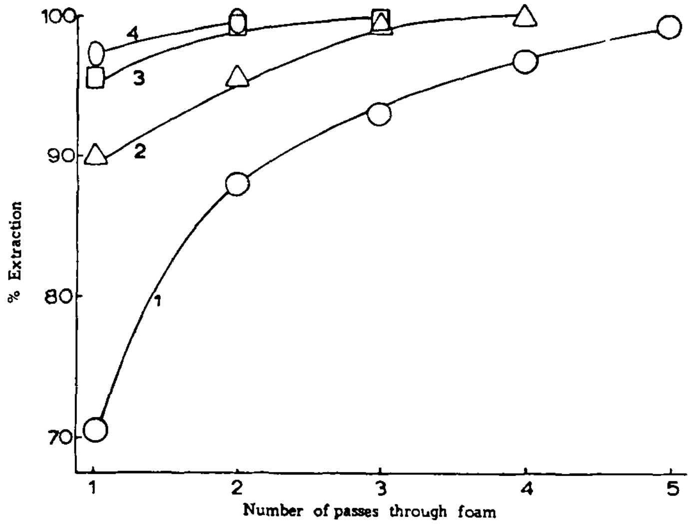

FIG. 5. Effect of number of foam plugs and passes on the extraction efficiency of gallium. Curve numbers refer to the number of foam plugs used. One hundred milliliters of 50 ppm of Ga in 7.5 *N* HCl was used at flow rate of 64 ml/min at room temperature.

the removal of gallium from solution or the recovery of gallium from the foam.

Under conditions of lower than 100% extraction of gallium by foam (e.g., 25°C and high flow rates of 64 ml/min) the efficiency can be increased either by passing the solution through several foam plugs or by passing the same solution through the foam several times. This is shown in Fig. 5 where the extraction efficiency at 25°C is determined for 1, 2, 3 and 4 foam plugs with 1 to 5 passes of the acid gallium solution through the foam plugs.

A foam plug which weighed about 1 g has a limited capacity. This is illustrated by the fall-off in extraction efficiency as the amount of gallium in the foam increases, shown in Fig. 6, where a standard solution (100 ml of 100 ppm Ga in 7.5 *N* HCl) was passed through a foam plug three times at a flow rate of 64 ml/min at 25°C and the extraction efficiency determined. This was repeated 13 times with fresh solution, allowing the gallium to accumulate in the foam. The efficiency falls from 100 to 50% when about 65 mg of gallium has been absorbed in the 1 g foam plug. It is expected

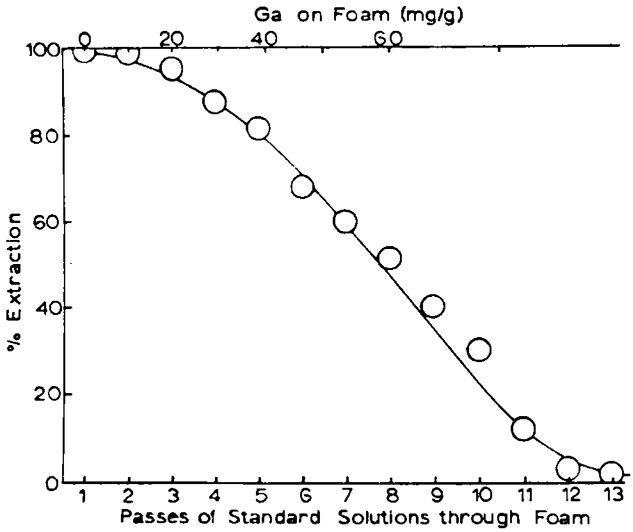

FIG. 6. Effect of Ga on the foam on the extraction efficiency for more gallium. One hundred milliliters of 100 ppm Ga in 7.5 *N* HCl was passed through a foam plug 3 times at a flow rate of 64 ml/min at room temperature. The extraction efficiency was determined and the process was repeated with another 100 ml of solution (13 times).

that this capacity would vary from one foam type to another and would depend on composition, structure, and density of the foam. Under optimum conditions it has been possible to absorb gallium in the foam to the extent of 10% by weight of foam.

These results suggests that the gallium chloride complex in acid solution dissolves in the foam. The very small surface areas determined for the foam, less than 1 m^2/g (*11*), confirms this.

The acid most often used to leach ores is sulfuric acid. Gallium in sulfuric acid solutions is not extracted by OCPUFS, although it is extracted to a small extent (>1%) by methyl ethyl ketone (*6*). However, when hydrochloric acid or sodium chloride is added to the sulfuric acid solution containing gallium, then the OCPUFS is effective in extracting the gallium from the solution. This is shown in Fig. 7 where the efficiency of extraction is plotted against the concentration of HCl in 50 ml of 1 : 2 H_2SO_4 : H_2O solution containing 50 ppm gallium. The results show that the addition of chloride to the sulfuric acid solution increases the efficiency with

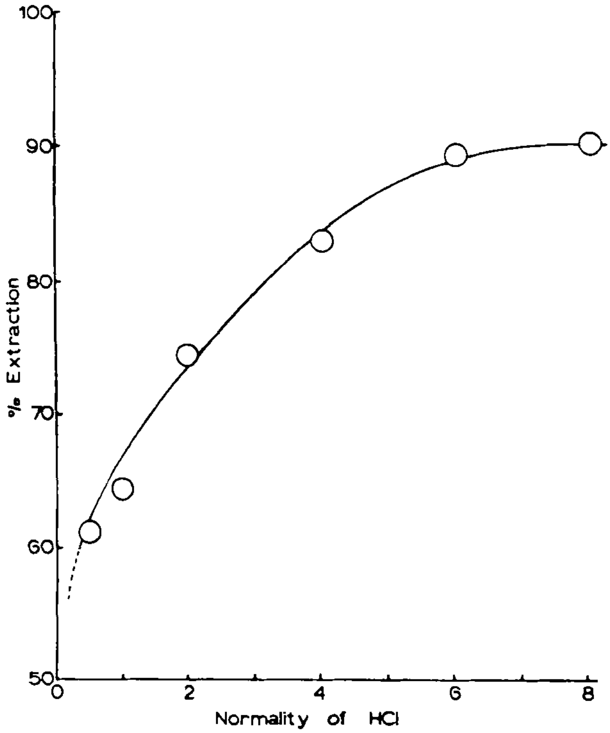

FIG. 7. Effect of HCl normality on extraction efficiency of Ga from a 1:2, H_2SO_4:water solution. Fifty milliliters of 50 ppm Ga in 10 *N* H_2SO_4 and various strengths of HCl was passed through a foam plug at a flow rate of 64 ml/min at room temperature.

which the foam extracts gallium from the solution. Similar results were obtained by Rafaeloff using methyl ethyl ketone as the extracting solvent (*6*).

Other metals as complex chlorides can also be absorbed by OCPUFS and could interfere with the absorption of gallium or reduce the capacity of the foam for gallium. We have shown that iron is one such element. In 7.5 *N* HCl the iron is not as efficiently absorbed by the foam as is gallium. This is shown in Fig. 8 where the iron capacity of a 1 g foam plug of up to 50% extraction efficiency is 44 mg/g of foam as compared to 65 mg/g foam for gallium.

Iron absorbed in the foam affects the efficiency of the foam for gallium extraction. This is shown in Fig. 9 for increasing amounts of iron in the foam. The drop-off in efficiency of gallium extraction as the amount of iron in the foam increases means that when the concentration of the iron in the solution is high, it may be desirable to remove it chemically from the solution prior to the removal of gallium by the foam.

Similarly the foam can be used to remove iron from acid solution.

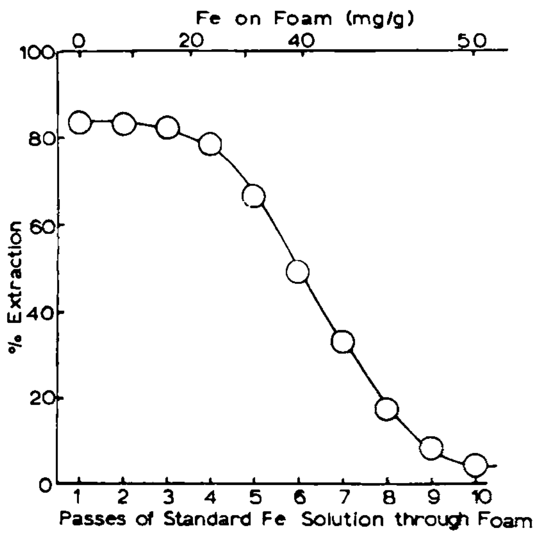

FIG. 8. Effect of iron on the foam on the extraction efficiency for more iron. One hundred milliliters of 100 ppm Fe in 7.5 *N* HCl was passed through a foam plug 3 times at a flow rate of 64 ml/min at room temperature. The extraction efficiency was determined and the process was repeated with another 100 ml of solution (10 times).

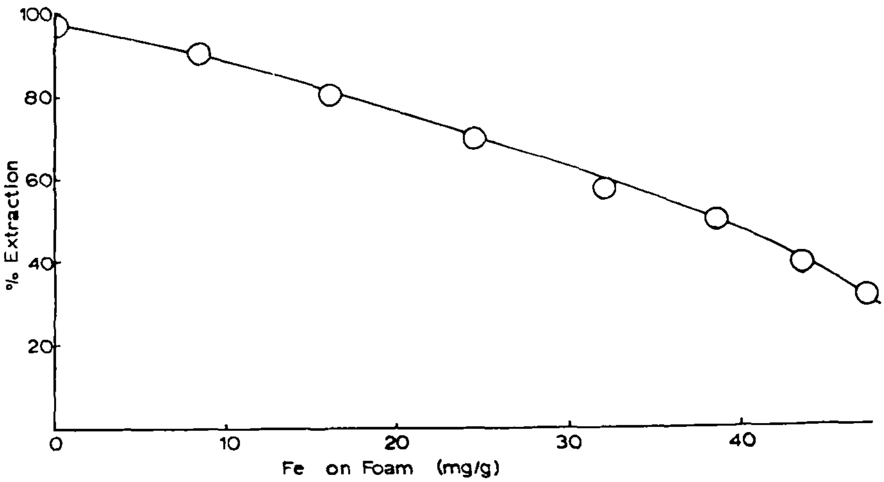

FIG. 9. Effect of iron on the foam on the extraction efficiency for Ga. One hundred milliliters of 100 ppm of Fe in 7.5 *N* HCl was passed through a foam plug 3 times at a flow rate of 64 ml/min at room temperature to give the increasing amounts of iron in the foam. Fifty milliliters of 50 ppm of Ga in 7.5 *N* HCl was passed through the iron-containing foam plug at a flow rate at 64 ml/min at room temperature and the extraction efficience for Ga was determined.

Aluminum at concentrations of over 1000 times more than gallium is not absorbed by the foam, neither does it interfere with the absorption of gallium.

CONCLUSIONS

The results clearly indicate that solid organic phases can act as "Solvent extractors." Open-cell polyurethane foam sponge is particularly suitable as a substitute for ether extraction systems when the polyurethane contains the polyether prepolymer. The kinetic and thermodynamic parameters of the foam as a true solvent extractor are now under investigation.

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