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Crosslinked Polyacrylamide Gel as a Dehydrating Agent

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Synopsis

The dried crosslinked gel prepared from acrylamide and *N,N'*-methylenebisacrylamide has been shown to be an efficient drying agent. The effectiveness of the dried gel has been shown to be comparable with that of several inorganic dehydration agents.

INTRODUCTION

Crosslinked polyacrylamide gels are finding increasing use in a wide variety of applications, ranging from grouting of mines and tunnels[1] to separation of components in human serum.

The gel, usually made by the polymerization of an aqueous solution of acrylamide and *N,N'*-methylenebisacrylamide, contains about 90% water and 10% crosslinked polymer. It is permeable by diffusion to a wide variety of water-soluble chemicals.[2] Because of its properties, including permeability, the gel has been used as an isolation and separation medium for antiserums,[3] for the separation of hemoglobin,[4,5] for the study of immune precipitates,[6] and in the electrophoresis of human serum[7] or of proteins.[8]

More recently, dried acrylamide gels have been used in separations of such diverse materials as bacterial RNA,[9] human serum,[10] pollen pigments,[11] and cytochrome C.[12]

Since the dehydrated gel should be a good absorber of water, and indeed had been noted to reswell to the original gel volume when immersed in water, it seemed quite probable that the dried gel would make a good dehydrating agent. In addition, the dried gel should offer some advantages such as being neutral, nonionic, and low in density as compared with other available dehydrating agents. The products most commonly used for dehydration are inorganic materials[13] such as sulfuric acid, phosphorus pentoxide, calcium oxide, calcium chloride, calcium sulfate, sodium sulfate, alumina, molecular sieves, etc. Comparisons were made between some of these reagents and the dried gel.

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EXPERIMENTAL

Preparation of Dried Acrylamide Gel

In a stainless steel container, fitted with a coil for cooling and a thermometer, 10 lb. of acrylamide–methylenebisacrylamide mix (90% acrylamide/10% methylenebisacrylamide) was dissolved in 40 lb. of water. The solution was filtered through a Sparkler filter to remove any insoluble matter. To the stirred solution was added 45 g. of ammonium persulfate and 45 g. of dimethylaminopropionitrile. In 8 min. a gel was formed, and agitation was halted. A 3/1 mixture of methanol to gel was blended in a Waring Blendor, and the ground gel filtered through a Buchner funnel. The gel particles were air-dried on trays. The dried gel was put through a micropulverizer to give 10 lb. of an off-white powder of dried, crosslinked polyacrylamide which was used in the experiments. The dried gel produced in this fashion contained 13.9% water as measured by placing a sample in an oven at 125°C. for 7 hr.

Measurement of Drying Efficiency

The method of Barnitt et al.[14] was used. A quantity of the drying agent to be evaluated was weighed into an evaporating dish. Copper sulfate ($CuSO_4 \cdot 5H_2O$) was weighed into a Petri dish. The evaporating dish and drying agent were placed in the bottom of a desiccator. The Petri dish containing the copper sulfate was placed on top of the plate in the desiccator. The desiccator was covered, and the change in weight of the copper sulfate with time was followed. Table I indicates the weight losses to be

TABLE I
Weight Loss in Dehydration of $CuSO_4 \cdot 5H_2O$

Step	Weight loss, %
$CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 \cdot 4H_2O$	7.22
$CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 \cdot 3H_2O$	14.43
$CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 \cdot 2H_2O$	21.65
$CuSO_4 \cdot 5H_2O \rightarrow CuSO_4 \cdot H_2O$	28.86

expected in the stepwise dehydration of $CuSO_4 \cdot 5H_2O$ to anhydrous $CuSO_4$. The experiments reported here show that the maximum dehydration by any of the agents studied involves the removal of two moles of water to form $CuSO_4 \cdot 3H_2O$ (14.4% weight loss). Results are presented in Figures 1 and 2.

The acrylamide gel sample, shown in Figures 1 and 2, could be reused after heating in an oven at 80°C. for 1 hr. or longer. Results obtained with such regenerated dried gels were superimposable on the rates depicted in Figures 1 and 2.

The ability of the dehydrated acrylamide gel to absorb water also was measured by allowing samples of the polymer to stand in a desiccator over water. The gain in weight with time was followed and is shown in Figure 3.

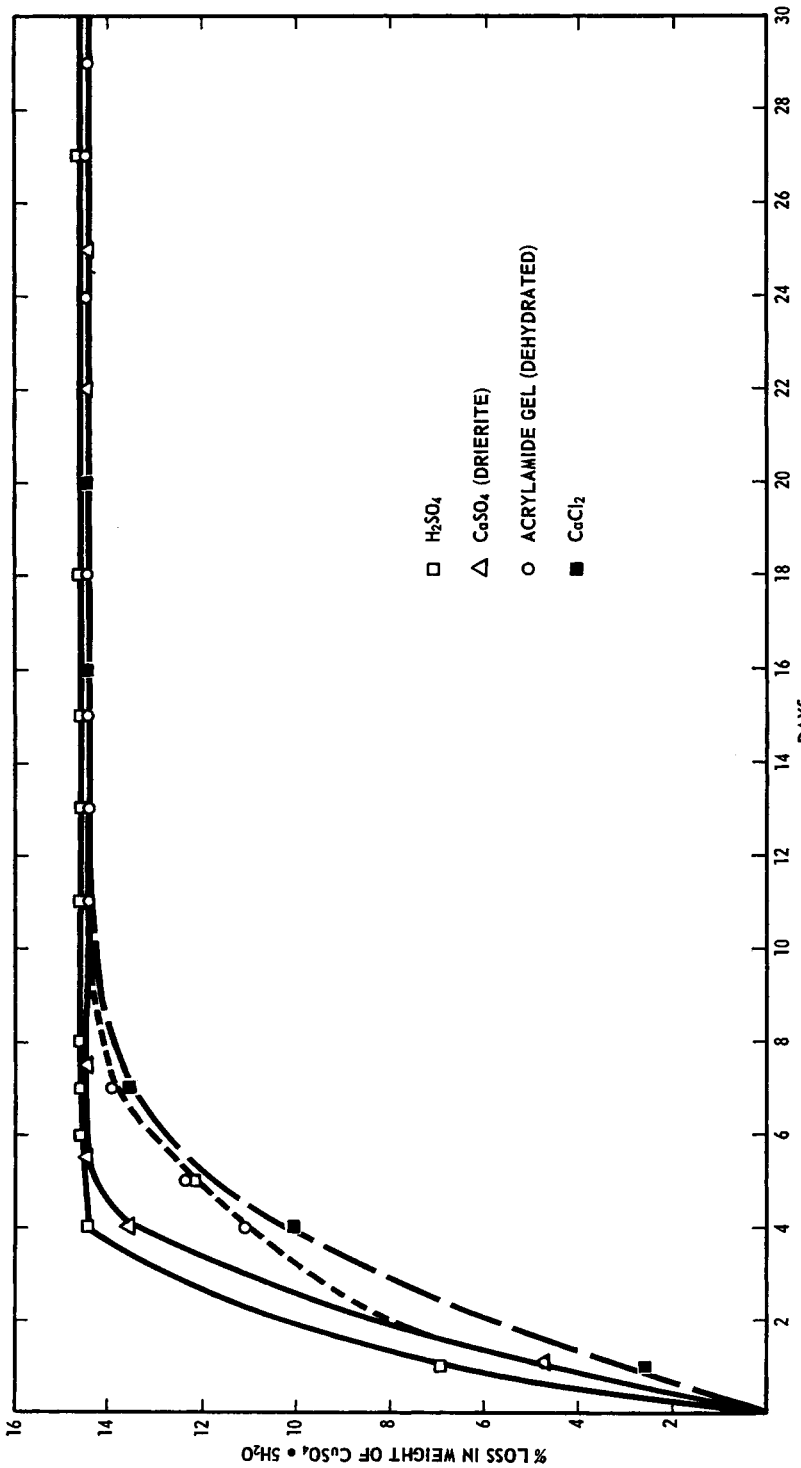

Fig. 1. Effectiveness of drying agents.

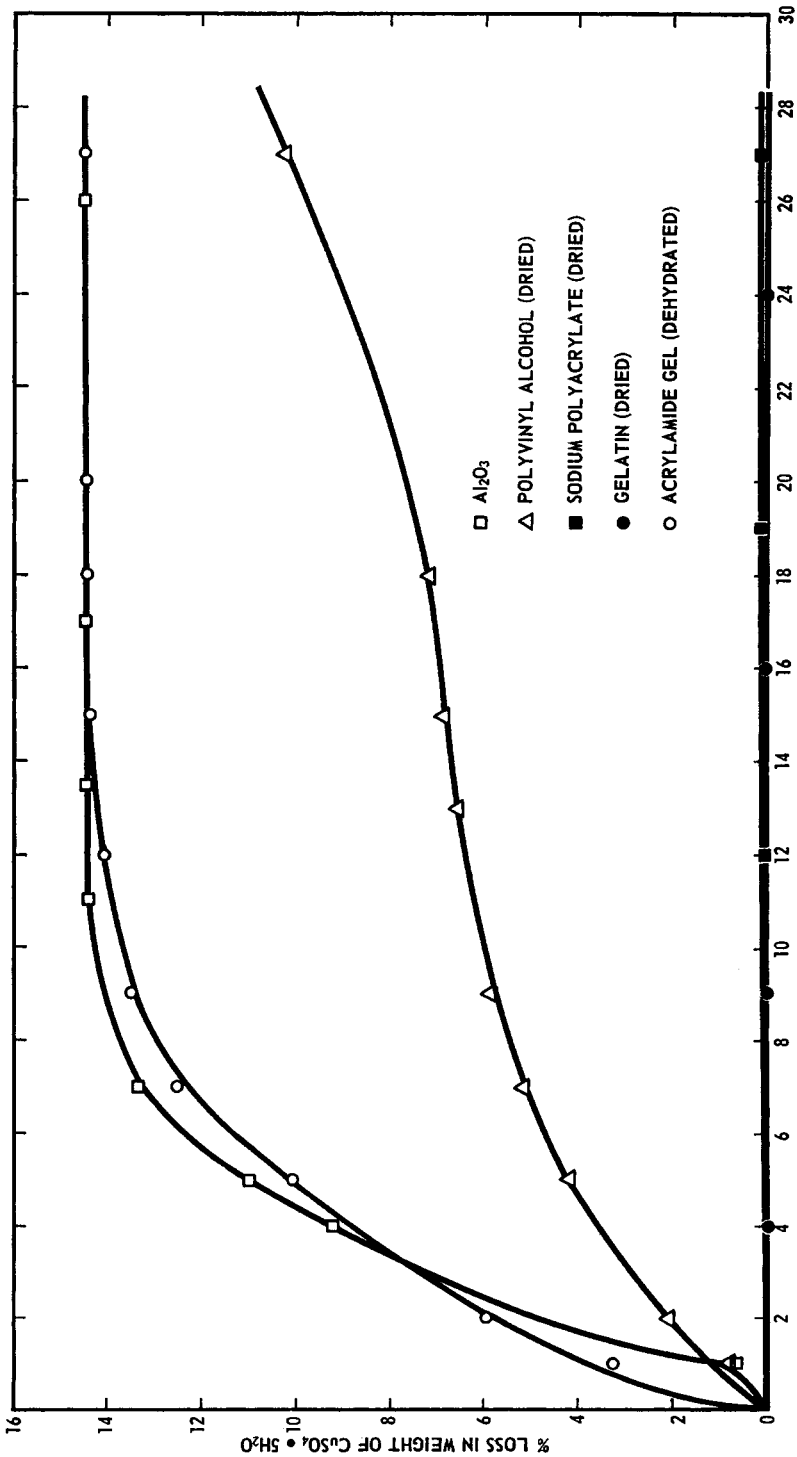

Fig. 2. Dried acrylamide gel as a drying agent.

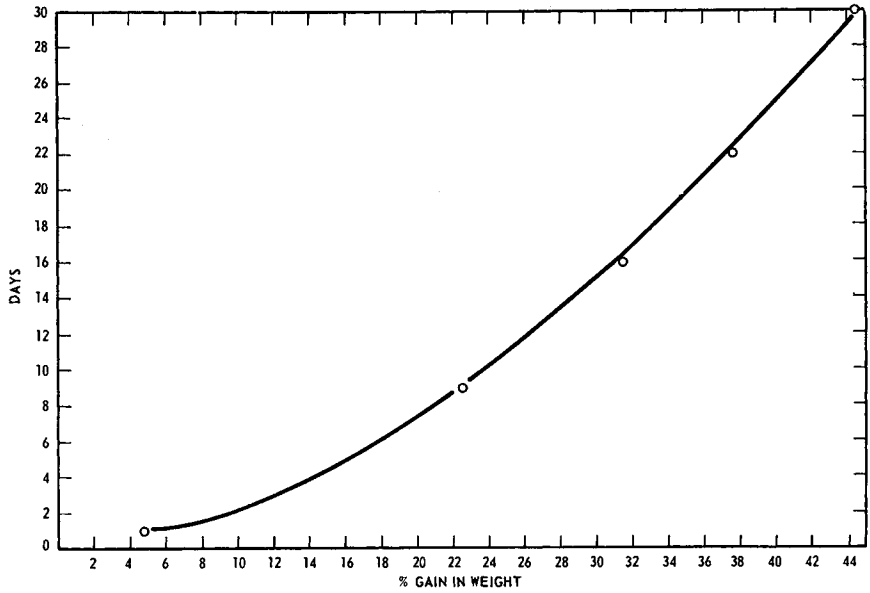

Fig. 3. Gain in weight of dehydrated acrylamide gel on standing over water.

After 30 days, the original free-flowing powder was converted to a spongy solid, having absorbed 0.445 g. water/g. original dried polymer.

In another test of dehydrating ability, 10 g. of benzene and 5 g. of water were placed in a bottle, 3 g. of crosslinked, dried acrylamide gel powder was added, and the bottle shaken. The crosslinked acrylamide appeared to absorb all the water and a benzene layer was observed above the swelled gel. Filtration of the mixture gave 6 g. of benzene and 12 g. of gelled mixture. The gel could be squeezed to obtain 3.9 g. of benzene.

DISCUSSION

The dried acrylamide–methylenebisacrylamide gel appears to be as efficient as several of the dehydrating agents tested during the 27 days of the evaluation. After one day, the anhydrous acrylamide gel seemed to be more efficient than either $CaCl_2$ (Fig. 1) or Al_2O_3 (Fig. 2) based on the water absorbed per gram of reagent. The anhydrous gel also was more effective than the other water-soluble polymers studied (Fig. 2). Dried gelatin or dried sodium polyacrylate did not remove any water from the $CuSO_4 \cdot 5H_2O$ under the conditions of the experiments.

The previous work of White and Dorion[2] has shown that the 90/10 acrylamide–methylenebisacrylamide gels swell less than either the 95/5 or 97/3 acrylamide–methylenebisacrylamide gels. Therefore, it might be expected that these latter two gels, which contain less crosslinks, when dried, could be more efficient as drying agents.

Perhaps part of the effectiveness of the gel in chromatographic or electrophoretic separations is related to its efficiency as a dehydrating agent. Water is absorbed very strongly into the brush pile structure of the polymer, and this, along with the many amide sites capable of hydrogen bonding, accentuates differences in the diffusion rates of different solutes. There is, of course, the added effect that certain large molecules cannot pass through the polymer interstices.

The gel is regenerable as a drying agent. It appears that the dried gel is worthy of evaluation in applications where a neutral, nonreactive, low density dehydration agent is required.

Mr. D. J. Wilson and Mr. J. Roshal kindly prepared the dried acrylamide gel which was used in these experiments.

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Résumé

Des gels séchés d'acrylamide et de *N,N'*-méthylene bisacrylamide sont des agents désèchants efficaces. L'efficacité de ces gels séchés est comparable à celle de nombreux agents deshydratants inorganiques.

Zusammenfassung

Das aus Acrylamid und *N,N'*-Methylenbisacrylamid dargestellte, getrocknete, vernetzte Gelist ein wirksames Trocknungsmittel. Die Wirksamkeit des getrocknten Gels ist derjenigen anorganischer Entwässerungsmittel vergleichbar.

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