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PURIFICATION OF AQUEOUS HYDROXYETHYLCELLULOSE. EXTRACTION OF SODIUM ACETATE WITH ISOPROPANOL AND HIGH-PRESSURE CARBON DIOXIDE *

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Introduction

In typical supercritical extraction (SCE) processes, a supercritical fluid (SCF) is used to dissolve selectively a weakly volatile component from a multicomponent mixture. In some cases, however, the SCF is a poor solvent for the component to be extracted. In such cases, the SCF cannot be used directly as a solvent but, as shown here, it can be used to induce phase separation, thereby enhancing the effectiveness of another more suitable solvent.

Near the critical point of a hydrocarbon solvent, solutions of hydrocarbon polymers exhibit a lower critical solution temperature (LCST). Thirty years ago, Freeman and Rowlinson (1) observed that upon heating, a homogeneous polymer solution may separate into two fluid phases. Such phase splitting can provide the basis of a separation operation. Similarly, it has been known for many years that injection of a high-pressure gas into a binary liquid mixture can induce a phase

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separation (2), as shown, for example, by Weinstock (3) for the system acetone/water/ethylene. At 50 bar and 15°C, water and acetone (and ethylene/acetone) are completely miscible. However, upon injection of ethylene into an aqueous solution of acetone, the ternary system can exhibit two liquid phases, in addition to a gas phase, at the same conditions of pressure and temperature.

More recently, McHugh and Guckes (4) reported phase separations in ternary polymer solutions. These authors found that addition of a supercritical fluid (SCF) to a binary polymer solution can shift the LCST to lower temperatures; the extent of the shift depends on the molecular weight of the polymer and on the solubility of the SCF.

We have extended the work of McHugh and Guckes toward development of a possible process for the purification of a water-soluble polymer: hydroxyethylcellulose (HEC), a cellulose ether with wide applications in the pharmaceutical, food and cosmetics industries. Annual consumption of HEC in the U.S. exceeds $30*10^3$ tons (5).

HEC is prepared by reaction of ethylene oxide with cellulose in aqueous alkaline solution. Sodium hydroxide is used to break the crystallinity of native cellulose, and also functions as a catalyst. The hydroxyethylated cellulose is soluble in water but insoluble in common organic solvents. In current processes, the by-product salt (from the neutralization of sodium hydroxide with acetic acid) and by-product glycols are extracted with aqueous solutions of alcohols or acetone. The washing process, including recycling of water and solvents, contributes significantly to the total cost of HEC (5).

In this work, we present an alternative purification method. By-product salt is removed upon inducing a phase split with supercritical carbon dioxide.

Experimental

Phase equilibria were measured for water-polymer-isopropanol-salt-gas mixtures for temperatures to 100°C and for pressures to 200 bar. The experimental apparatus, shown in Figure 1, consists of three sections: high-pressure equilibrium cell, feed system, and phase sampling.

The equilibrium cell is a high-pressure stainless-steel Jerguson liquid-level gage, approximately 150 cm^3, with two quartz windows for visual observation. Mixing in the equilibrium cell is achieved by recirculating both phases; the top phase is drawn from the top of the cell, circulated through a VICI six-port switching valve (5 in Figure 1), and then driven back to the bottom of the cell by a Milton-Roy metering simplex minipump (3). The bottom phase is drawn from the bottom of the cell, circulated through a VICI six-port switching valve (4) and then through a Mettler-Paar vibrating-tube densimeter before it is driven back to the top of the cell by a pump (2). The equilibrium cell and sampling valves are located in a constant-temperature air-bath, where the temperature is monitored and controlled by an Omega-Engineering heater-controller. The temperature of the densimeter cell is kept constant with an external oil bath. The pressure in the system is measured with a Heise Bourdon-tube pressure gauge. Estimated accuracies of the pressure and temperature measurements are 0.2 bar and 0.1 K, respectively. The volume of the sampling loops are approximately 1.3 cm^3; the exact value depends on the temperature. At each temperature, volume calibration is accomplished by sampling a saturated salt-water solution of known composition and density.

The feed system includes a CO_2 gas cylinder, a condenser kept at 5°C for liquefaction of CO_2, and a metering pump for liquid CO_2. A stainless-steel tank feeds the polymer solution to the cell; a few bars of nitrogen push the viscous solution to the cell through a 1/4-inch pipe.

We present results for two kinds of experiments. For some experiments, the polymer was placed in the system as a solid. For other experiments, the polymer was dissolved in water before feeding to the system. Since commercially available

HEC contains some salt, the polymer used for both kinds of experiments also contained salt.

In the first experiments, the objective is to measure the solubility of polymer in supercritical CO_2. A known amount of polymer is placed in a cartridge (7). After flowing CO_2 through this cartridge for several hours, the pressure is released; the contents of the cartridge are then weighed to detect possible changes in the amount of polymer. On-line filters (6) prevent solids from leaving the cartridge. In this case, the equilibrium cell plays the role of a solvent tank having an internal volume much larger than that of the cartridge.

In the second experiments, the objective is to study phase equilibria for the system water/polymer/salt/gas/isopropanol as a function of temperature, pressure, and overall composition. A known amount of aqueous polymer/salt solution is fed to the cell. Later, a known amount of isopropanol is also fed to the cell. Finally, CO_2 is pumped to the system until the desired pressure is attained. At a given thermodynamic state, the different phases present are observed through the cell windows. To ensure equilibrium, both recirculation pumps are operated for periods of over 15 hours ; when equilibrium is attained, the density displayed by the densimeter remains constant, and equilibrium phases have a homogeneous appearance. Having reached equilibrium, samples of the bottom and top phases are collected through the sampling valves.

Before each experiment, the equipment (cell, all lines, feed tank) is soaked with deionized water for more than two hours. This rinsing procedure is repeated four times, to ensure that no polymer or salt residues are left in the apparatus.

Figure 2 shows a diagram of the sampling section of the apparatus. Samples are trapped in a sampling loop of calibrated volume. When the sampling valves are switched, the contents of the loop are expanded to atmospheric pressure. The amount of CO_2 is measured by displacement of a CO_2-saturated aqueous solution. Gas volumes can be measured with an accuracy of 1%. Having evacuated the CO_2 from the sample, the sampling lines are soaked with deionized water for a period of two hours. Later, the lines are rinsed thoroughly with deionized water and the contents of the loop are collected.

Salt concentrations are measured with a Perkin-Elmer 2280 atomic absorption spectrophotometer. High-pressure liquid chromatography is used to measure the concentrations of polymer and isopropanol. Since the salt used for our experiments interferes with the polymer concentration measurements, a Biorad Aminex carbohydrate HPX-87C anion exchange column is used to trap the salt. A Biorad TSK40 Progel size-exclusion column, thermostated at $40°C$, is used to separate the isopropanol from the polymer. The detector is a Knauer differential refractometer connected to an integrator. Salt, polymer, and co-solvent concentrations are reproduced with a precision of about 1%.

Densities are obtained from the measured period of oscillation of a vibrating U-shaped sample tube through which the sample flows continuously. The density, ρ, is related to the period of oscillation, τ, by

$$\rho = A\,\tau^2 + B$$

where A and B are temperature-dependent instrument constants, determined by calibration with fluids of known density. In this work, we used pure CO_2, pure water, pure ethylene glycol and a saturated sodium sulfate solution to calibrate the densimeter. This array of substances covers a range of densities of about 0.5 g/cm^3. Densities are measured with an accuracy of 0.0001 gcm^{-3}.

Once the amounts of CO_2, polymer, salt, and isopropanol are known, the amount of water in a sample is calculated from the measured density and the known volume of the sampling loop.

Our experimental apparatus and procedure have several limitations. Since we only have one densimeter, only the density of one of the equilibrium phases can be measured; therefore the concentration of water can be measured only for that phase for which the density is known. A second, perhaps more severe limitation, is that the sampling lines that draw fluids from the equilibrium cell (and through the sampling valves) have a fixed position inside the cell. In a multi-phase mixture, we can only sample those two phases that happen to cover completely the inlets of these sampling lines.

Properties of HEC

In HEC, the hydroxyl groups of cellulose have been partially substituted with hydroxyethyl groups by reaction with ethylene oxide. Figure 3 shows the repeating unit of HEC. The properties of HEC are significantly influenced by the degree of substitution (DS) which designates the average number of hydroxyl positions on the anhydroglucose unit that have reacted with ethylene oxide. In all our experiments, we use HEC with a DS of three. The hydroxyethyl groups give HEC a polar character; as a result, HEC is soluble in water (and some other highly polar solvents) but insoluble in common organic solvents such as alcohols, ketones, ethers or hydrocarbons. The HEC used in our work has a molecular weight of about $7*10^4$. It was supplied by Union Carbide Corporation; its commercial name is Cellosize QP-09L.

Results

At moderate temperatures and pressures, electrolytes are insoluble in supercritical CO_2. Some polymers, however, are relatively soluble in supercritical solvents (6). For example, the solubility of polyethylene glycol (PEG) (which has some similarities to HEC) in CO_2 is about 0.5% by weight at $60°C$ and 150 bar (7). However, while PEG is liquid at room temperature, HEC is solid to $180°C$; above this temperature, it decomposes.

If HEC were soluble in CO_2, that solubility could be the basis for a salt-removal process. The first step in our experiments was to determine whether HEC is soluble or not in CO_2. Using the experimental method described above, we made measurements from 20 to $100°C$, and from 50 to 200 bar. For some of these experiments CO_2 was liquid. For all the conditions studied here, HEC was essentially insoluble in CO_2.

To determine if a polar entrainer could improve the solubility of the polymer, we also made some solubility measurements for solid HEC, in water-saturated CO_2.

Since HEC is hygroscopic, the water from the CO_2 was trapped by the polymer. After several hours, the contents of the cell were removed and dried. No polymer had dissolved in the moist carbon dioxide.

For all the experiments described below, the feed polymer solution is a 93.5 g/l solution of HEC in water. The HEC used for all of our experiments contains 4.3 weight % by-product sodium acetate, i.e. our polymer solution contains 4.2 g/l sodium acetate.

At 200 bar and 75° C, the solubility of CO_2 in water is only about 5 g/l (8). The presence of dissolved polymer in the water does not affect significantly the solubility of CO_2. Figure 4 shows the measured solubilities of CO_2 in the HEC solution, at different pressures and temperatures; these solubilities are almost identical to those reported by Gillespie and Wilson for polymer-free water (8). The data at 27°C are for a liquid-CO_2 phase in equilibrium with a liquid-water phase. For higher temperatures, the CO_2 phase is supercritical. No polymer was detected in the CO_2-rich phase for any of the experimental conditions shown in Figure 4.

The solubility of CO_2 in the polymer solution is not high enough to produce phase separation. If a phase split is to be induced by CO_2, we must increase its solubility by adding an organic co-solvent such as isopropanol.

Isopropanol/water mixtures are common in industry; the properties of these mixtures are well documented (9). An alternative co-solvent choice is ethylene glycol, which is a by-product in HEC manufacturing. However, less information is available for the CO_2/ethylene glycol/water system than for the CO_2/isopropanol/water system. Further, under some circumstances ethylene glycol may experience undesirable polymerization reactions that tend to plug the sampling lines of our experimental equipment.

At ambient conditions and in the absence of CO_2, isopropanol and water are miscible in all proportions. Higher alcohols (e.g. butanol) exhibit partial miscibility with water (10). Upon addition of CO_2, two phases (or three, depending on the temperature) are formed. For temperatures below 30°C and pressures above 75 bar, three phases can coexist: a CO_2 gas phase, a CO_2-rich liquid phase, and a water-rich

liquid phase. Propanol distributes between the two liquid phases (11). For temperatures above 35°C, only two phases coexist: a fluid CO_2-rich phase, and a water-rich phase. Again, propanol distributes between these two phases (9).

When HEC is present in solution, however, the phase behavior of the system is significantly different from that of the ternary $CO_2/C_3H_7OH/H_2O$ mixture. Figure 5 shows the solubility of CO_2 in a mixture of aqueous isopropanol/polymer solution as a function of pressure. The data in Figure 5 are for different ratios of alcohol to water in the mixture. For low proportions of alcohol, HEC does not precipitate out of solution; in the absence of CO_2 such mixtures are homogeneous. At 40°C and pressures below 40 bar, addition of isopropanol significantly raises the solubility of CO_2 with respect to that in the absence of alcohol. However, when the pressure rises isothermally to about 70 bar, the CO_2 and isopropanol concentrations in the aqueous polymer-rich phase drop dramatically because a second isopropanol/water/CO_2 liquid phase appears. As shown in Figure 6, reductions in CO_2 solubility are accompanied by reductions in isopropanol concentration.

As the pressure is further increased, the relative proportions of polymer-rich and isopropanol-rich phases vary. Between 80 and 90 bar, the isopropanol/water/CO_2 liquid phase disappears again, and only two phases are left in the system: a fluid CO_2-rich phase and a liquid polymer-rich phase. At constant temperature, as the overall alcohol-to-water ratio rises, there is an increase in the range of pressures where two liquid phases coexist.

The high viscosity of the polymer-rich phase makes difficult the precise measurement of the pressures at which these phase transitions take place. Also, for a given temperature, phase separation occurs at different pressures, depending on the relative proportions of water and isopropanol; for our experiments, these proportions must be measured visually and are therefore subject to some inaccuracy. There are, however, indirect methods for determining the range of pressures where two liquid phases coexist. Figure 7 shows the density of the polymer-rich phase as a function of pressure, at 40°C, for an alcohol/polymer-solution volume ratio of 2/5. For low pressures, the density increases slowly with pressure. At 70 bar, there is a sharp

increase in density. Above 80 bar, this density stabilizes again. Propanol has a density which is about 20% lower than that of water. When the second liquid phase appears, propanol comes out of the polymer-rich phase, thereby causing a density increase. When the second liquid phase disappears again, the amount of propanol in the polymer phase varies only slightly with pressure, and again the density increases slowly with pressure. The range of pressures where two liquid phases coexist corresponds to the vertical region of the S-shaped curve in Figure 7.

As the ratio of isopropanol to water rises, the two liquid phases form at lower pressures. For example, for a 35/50 (volume ratio) mixture of isopropanol and polymer solution, two liquid phases appear at 65 bar. Table I gives the number of phases present in the system for different pressures, temperatures, and overall compositions.

When the concentration of isopropanol exceeds a threshold value of about 30 volume %, the water/alcohol mixture is no longer a good solvent for the polymer, and HEC precipitates out of solution. If, however, the pressure rises sufficiently to form two liquid phases, all of the solid polymer redissolves in the water-rich phase.

Salt-Partitioning

For those experiments where the CO_2-rich or the isopropanol-rich phases were sampled, no polymer was detected in the samples. HEC only dissolves in aqueous solutions; if the alcohol content of a polymer-rich phase is too high, HEC precipitates out of solution but does not dissolve in the other coexisting fluid phases.

The situation for the salt, however, is different. Figure 8 gives the ratio of salt to polymer in the polymer-rich phase at $40°C$ as a function of pressure. The data shown in this figure are for different overall alcohol-to-water ratios. The dotted line indicates the initial salt/polymer ratio before the experiments, i.e. the ratio for commercial HEC. At high pressures, this ratio remains constant, regardless of the overall alcohol-to-water ratio. It remains constant because no salt or polymer go to the supercritical CO_2-rich phase.

In the region around 70 bar, depending on the relative proportions of alcohol and water, the salt/polymer ratio can decline. The range of pressures where these declines occur corresponds to that for the formation of two liquid phases (see also Figures 5 and 6). While sodium acetate partitions between coexisting liquid phases, all the polymer stays in the bottom, polymer-rich phase. This is a fortunate result for practical applications.

Larger reductions of salt concentration are observed for lower overall alcohol/water ratios than for higher alcohol/water ratios. Table II reports the compositions of different phases, at $40°C$, for different pressures. If we assume that large dielectric constants are responsible for salt solubility, then salt solubility is enhanced by large water to alcohol ratios. For low pressures and small propanol loads, the CO_2/isopropanol/water phase contains a significant fraction of water, while for higher pressures and larger propanol loads, the fraction of water is reduced.

Our measurements indicate that at $40°C$ and for 66 bar, for a 35/50 volume ratio of isopropanol to polymer-solution, the salt content of commercial HEC can be reduced about 30% in one stage. This result suggests that the use of CO_2 to induce a phase separation could provide the basis for a large-scale HEC purification process.

Conclusions

The results presented here show that at $40°C$ and 66 bar, supercritical carbon dioxide induces a liquid-liquid phase separation in aqueous mixtures containing about 8 wt% HEC, 0.25 wt% sodium acetate and isopropanol concentrations around 30 wt%. Since salt distributes between the two liquid phases, whereas HEC does not, liquid-liquid extraction could be used as the basis for a salt-removal process.

The phase-splitting effect of CO_2 may also have potential applications for other parts of current HEC manufacturing processes in the absence of salts; upon injection

of CO_2, small amounts of HEC are enough to promote the formation of an isopropanol-rich liquid phase and a water-rich phase. This phase behavior could be used for efficient recycle of isopropanol and water and for recovery of other by-products such as ethylene glycol.

Acknowledgments

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Table I - Number of phases in the system for different pressures, temperatures, and overall compositions.

isopropanol/water (volume ratio)	P (bar)	T (°C)	Number of Phases	Phases
20/50	71 (and below)	40	2	CO_2 gas polymer-rich liquid
20/50	77	40	3	CO_2 gas $CO_2/i-C_3$/water polymer-rich liquid
20/50	82 (and above)	40	2	CO_2 fluid polymer-rich liquid
60/40	40 (and below)	40	3	solid polymer CO_2 gas polymer-rich liquid
60/40	86.5	40	3	CO_2 gas $CO_2/i-C_3$/water polymer-rich liquid
60/40	100 (and above)	40	2	CO_2 fluid polymer-rich liquid
35/50	58	25	4	CO_2 liquid CO_2 gas polymer-rich liquid solid polymer

Table II - Composition of the aqueous polymer-rich phase for several pressures, temperatures, and overall compositions

Overall Volumetric Ratio $i-C_3/H_2O$	P (bar)	T (°C)	w_{HEC} $*10^2$	w_{NaAc} $*10^3$	w_{i-C_3}	w_{CO_2} $*10^2$	w_{H_2O}	Density (g/cm^3)
20/50	79	40	6.44	2.66	0.215	5.53	0.662	0.9831
35/50	67	40	7.92	2.38	0.294	6.79	0.556	0.9631
60/50	77	40	9.79	3.63	0.224	5.68	0.618	0.9870
50/50	154	40	6.90	3.04	0.186	5.30	0.690	0.9887

All concentrations are reported in weight fraction.

FIGURE 1

Experimental apparatus. (1) Feed pump. (2) Recirculation pump, bottom phase. (3) Recirculation pump, upper phase. (4) Six-port sampling valve, bottom phase. (5) Six-port sampling valve, upper phase. (6) 15-μm filter to prevent solid polymer from leaving the cartridge. (7) Cartridge for solid polymer.

FIGURE 2

Sampling section. After taking a sample, the amount of gas is measured by liquid displacement. The remaining solution is flushed out with a known amount of deionized water and collected for analysis.

FIGURE 3

Molecular formula of the repeating unit of hydroxyethylcellulose.

FIGURE 4

Solubility of carbon dioxide in the aqueous polymer-rich phase as a function of pressure (in the absence of propanol) at 27, 50, 75 and 100°C.

FIGURE 5

Solubility of carbon dioxide in the aqueous polymer-rich phase, as a function of pressure, at 40°C, (a) in the absence of propanol and (b) in the presence of propanol. The decrease in CO_2 concentration below 70 bar, in the presence of propanol, corresponds to the formation of two liquid phases.

FIGURE 6

Concentration of propanol in the aqueous polymer-rich phase as a function of pressure at 40°C. The data shown are for several overall propanol/water volume ratios. The decrease in propanol concentration corresponds to the formation of two liquid phases.

FIGURE 7

Density of the aqueous polymer-rich phase as a function of pressure at 40°C. The overall propanol/water volume ratio is 20/50. The vertical region of the curve corresponds to the formation of two liquid phases.

FIGURE 8

Salt/polymer ratio in the polymer-rich phase, as a function of pressure, at 40°C. The data shown are for several propanol/water overall volume ratios. The dotted line represents the salt/polymer ratio of commercial HEC.

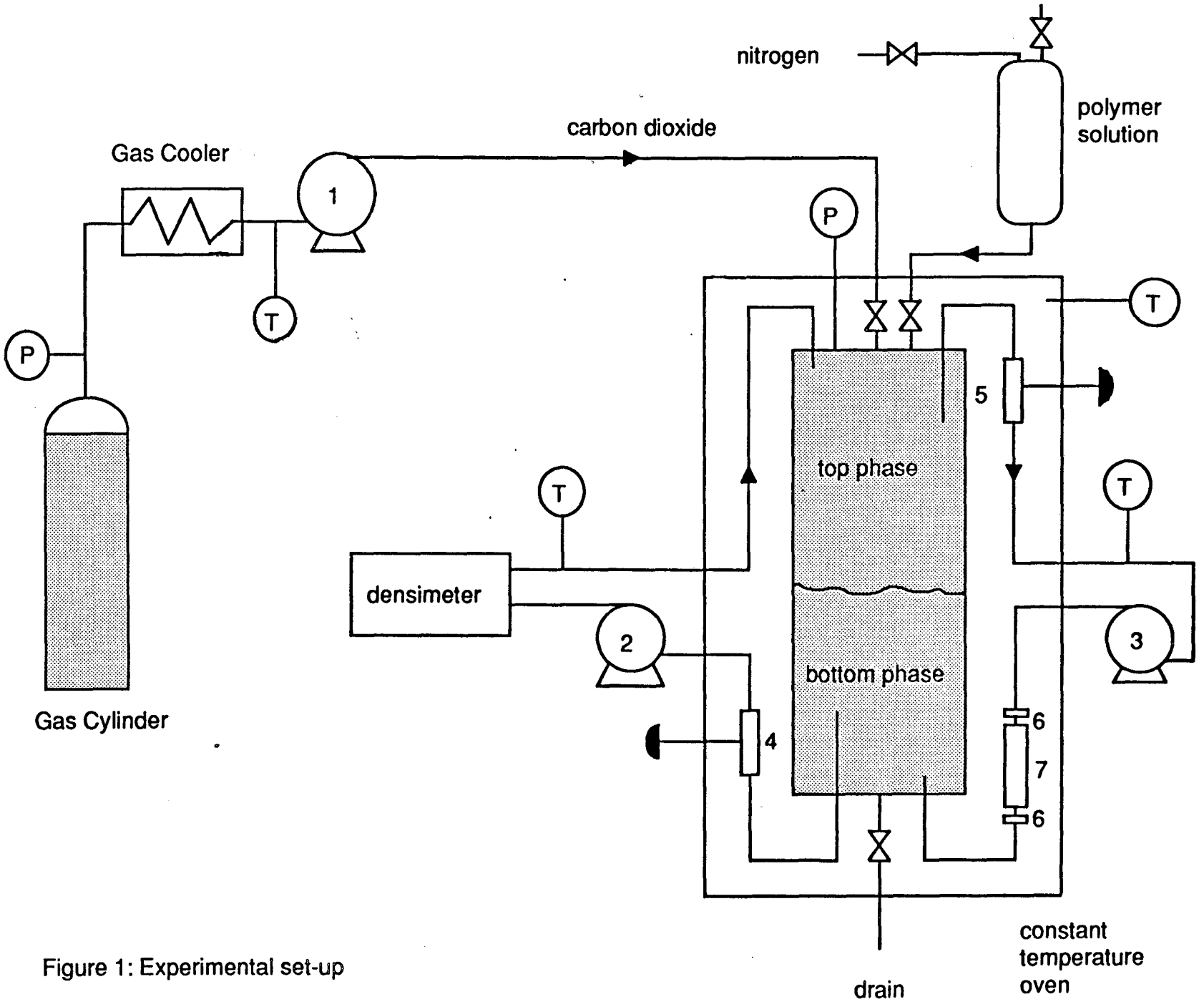


Figure 1: Experimental set-up

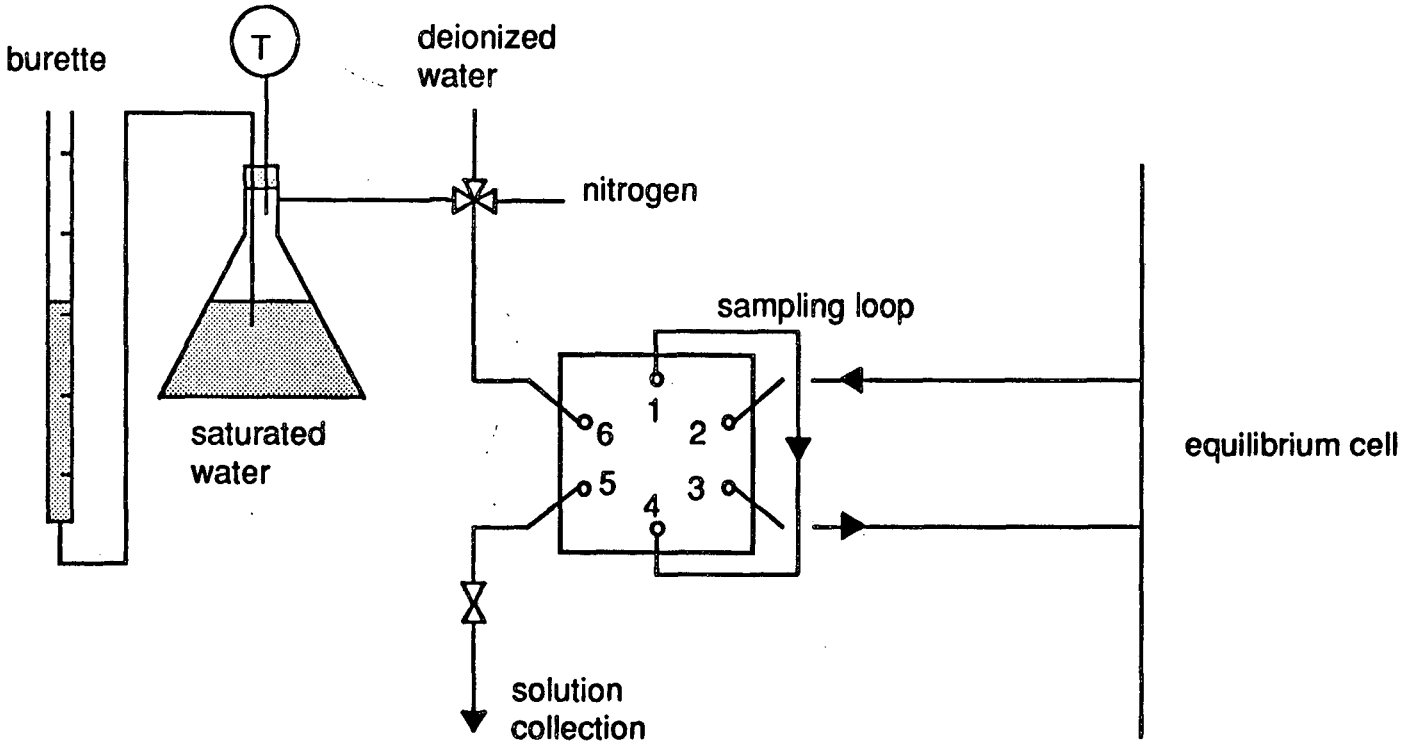
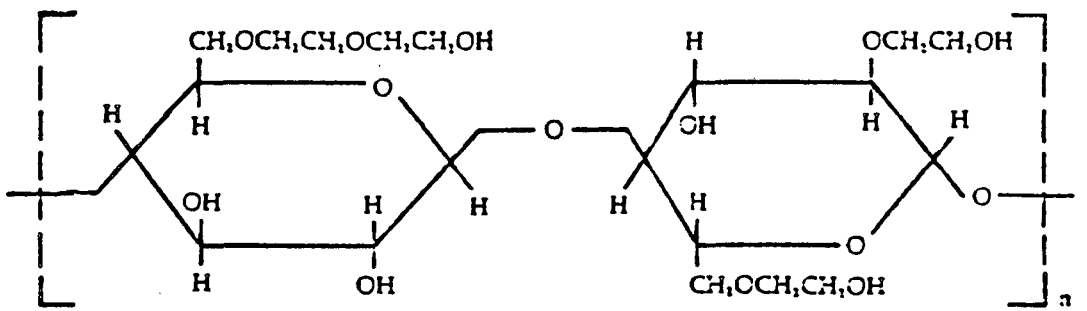


Figure 2 : Sampling section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H
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OCH₂CH₂OH
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FIGURE 3

SOLUBILITY OF CARBON DIOXIDE IN THE AQUEOUS POLYMER-RICH PHASE IN THE ABSENCE OF PROPANOL

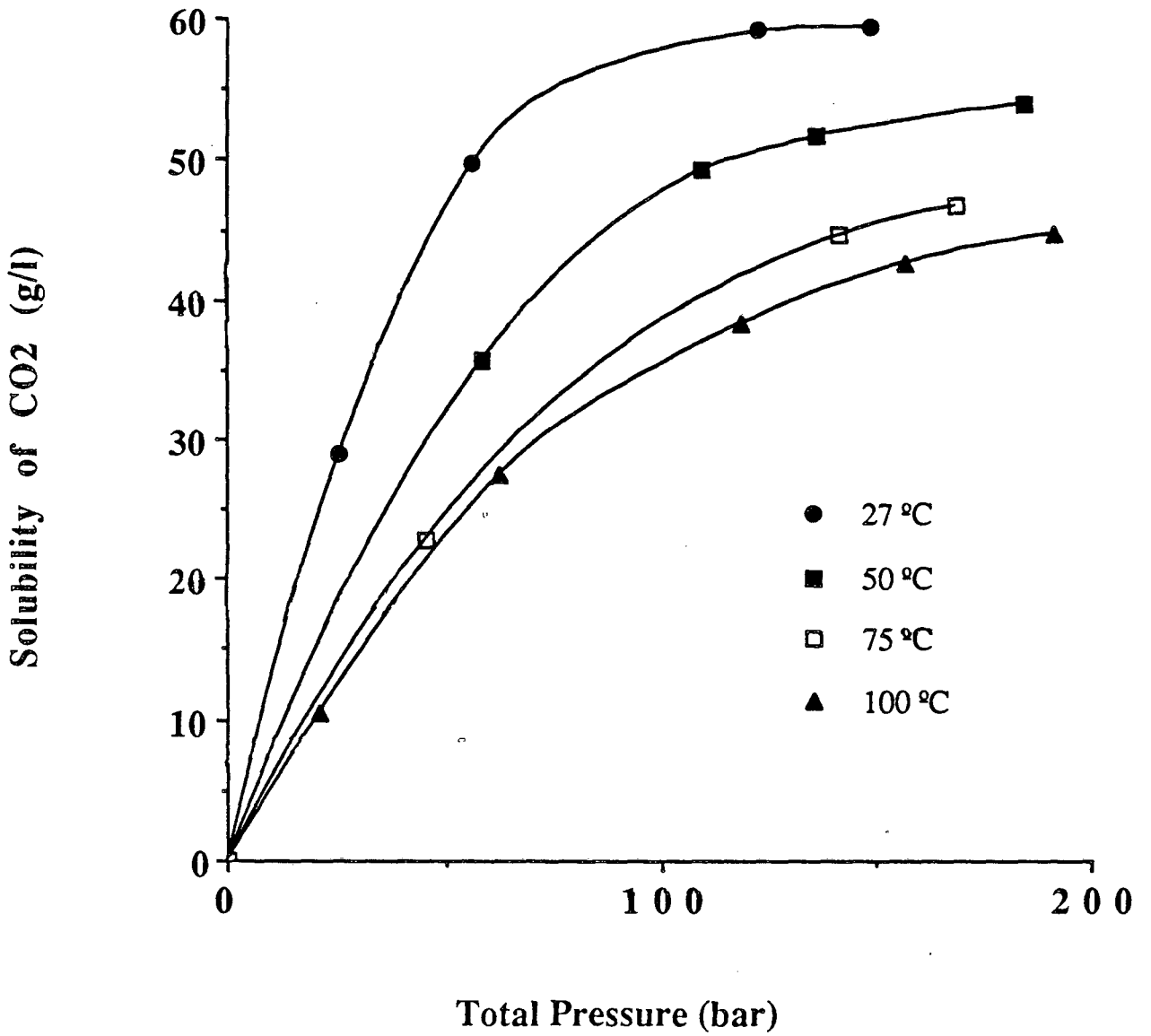
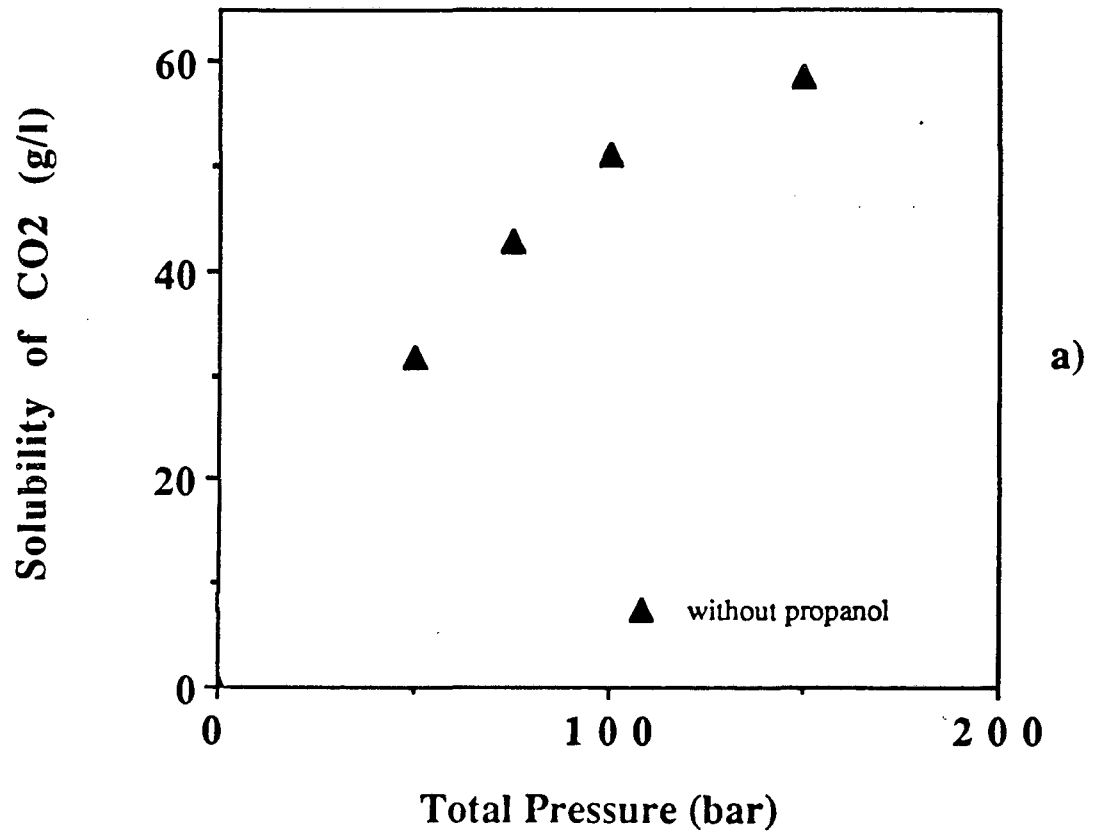


FIGURE 4

SOLUBILITY OF CARBON DIOXIDE IN THE AQUEOUS POLYMER PHASE AT 40ºC



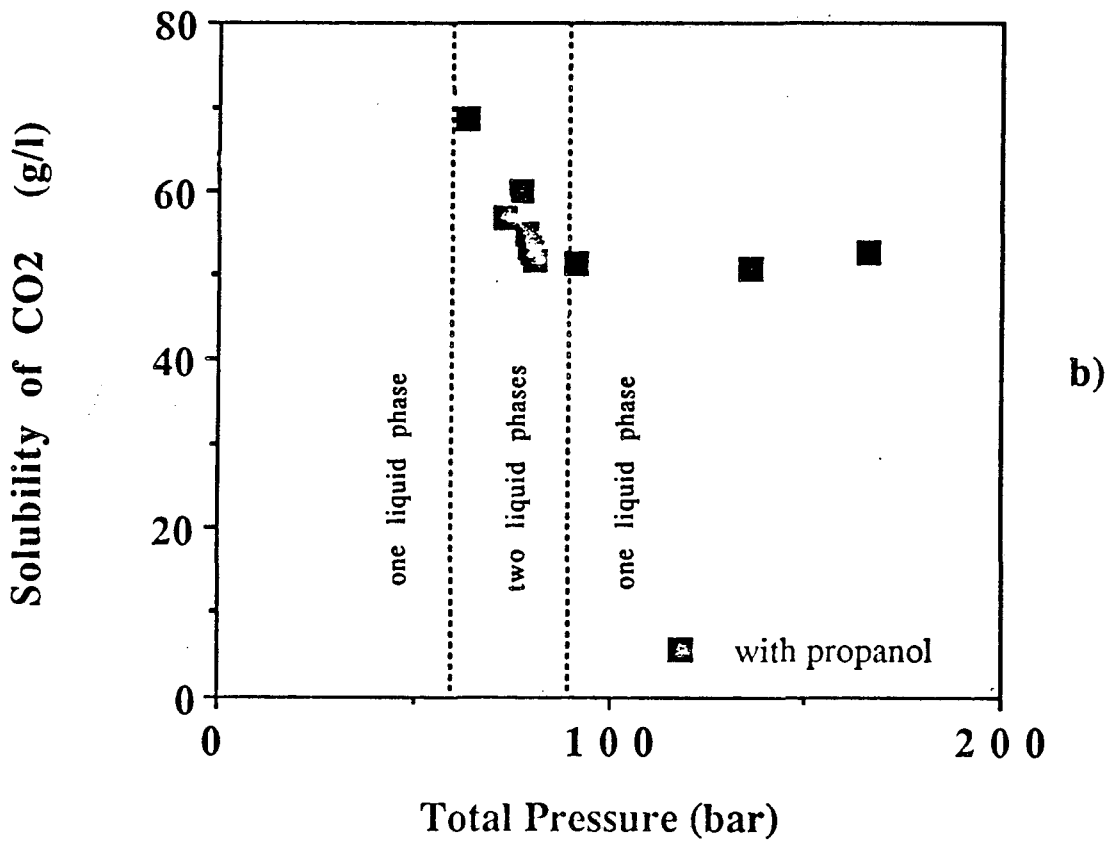


FIGURE 5

SOLUBILITY OF PROPANOL IN THE AQUEOUS POLYMER-RICH PHASE AT 40 ºC

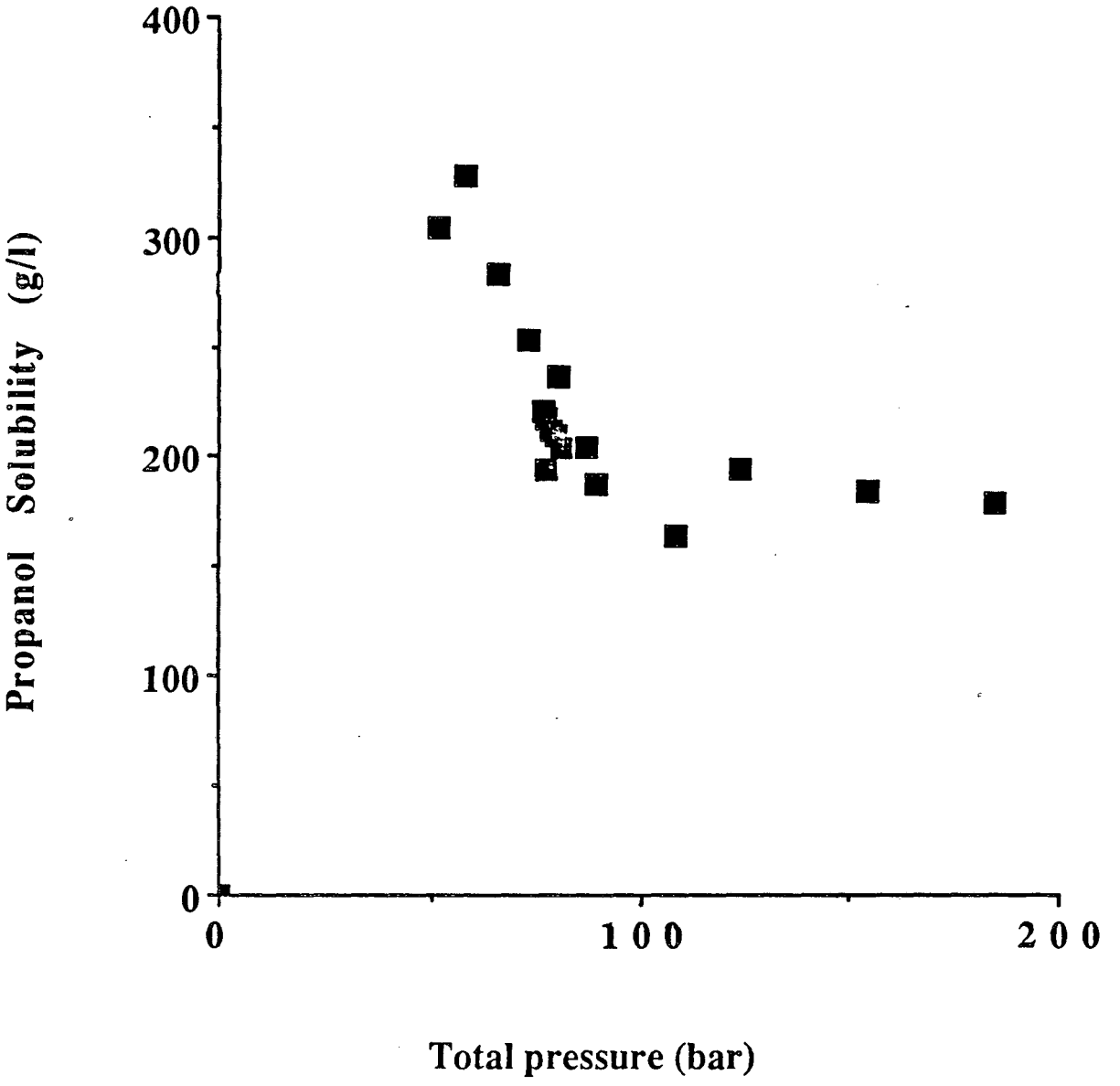


FIGURE 6

DENSITY OF THE AQUEOUS POLYMER-RICH PHASE AT 40 ºC

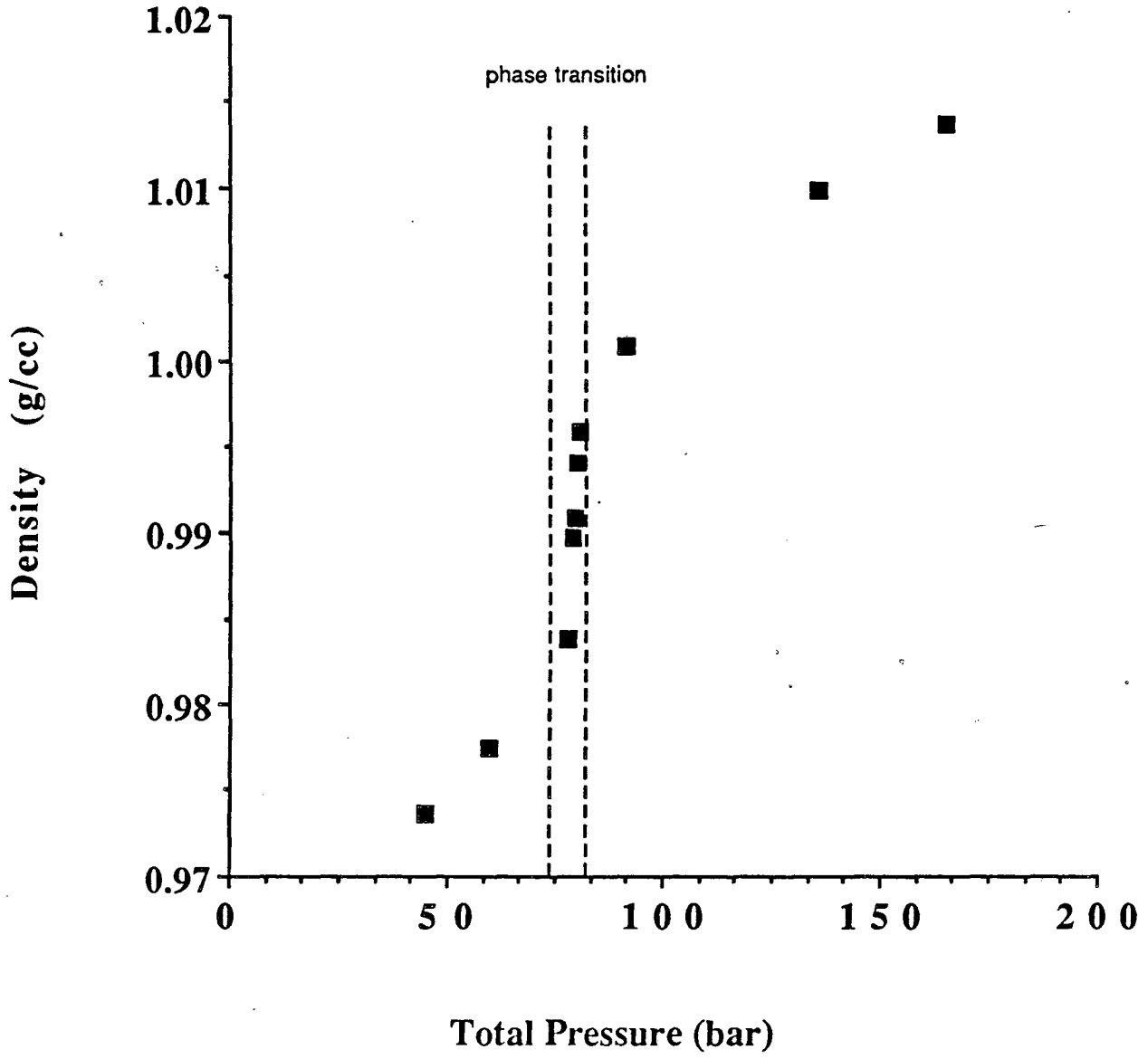


FIGURE 7

SALT/POLYMER RATIO IN THE AQUEOUS POLYMER-RICH PHASE AS A FUNCTION OF PRESSURE AT 40 ºC

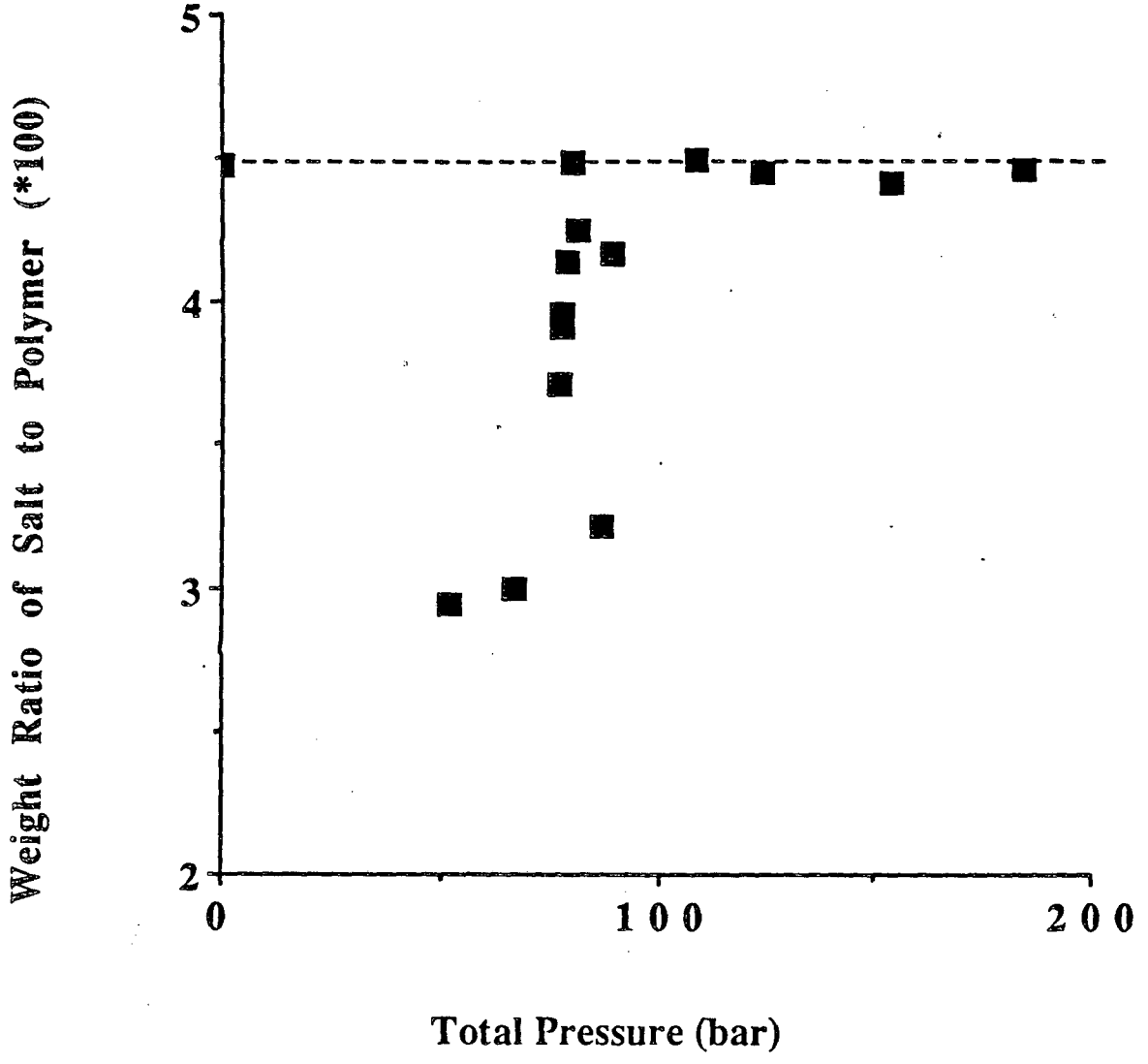


FIGURE 8

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