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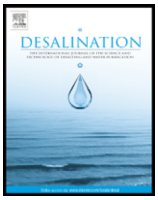

The breaking down of the 2-butanone/water/*n*-butanol/sodium dodecylsulfate microemulsion system by pervaporation technique: Effect of the stirrer rate

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ABSTRACT

The principle that stipulates the removal of a certain quantity of one of the microemulsion components leads to destabilize and break down a microemulsion. This work investigated the breaking down of two microemulsions by pervaporation technique: 2-butanone-rich and water-rich microemulsions. Poly(dimethylsiloxane) was used as a hydrophobic membrane for extracting 2-butanone from the oil-rich microemulsion system and poly (vinylalcohol) was used as a hydrophilic membrane for the removal of water from water-rich microemulsion. The pervaporation parameters, the total flux rate and the enrichment factor, were examined as a function of stirrer rate, time and temperature. During these operations a turbidity was observed in the feed, indicating that the microemulsion was broken down. It was observed that more marked breaking points of these microemulsions were obtained at the lower stirrer rates. At 60 °C, 2 h was sufficient for breaking down the microemulsions and extracting 9.0 ± 0.2 g (~13 wt.%) of 2-butanone and 8.5 ± 0.2 g (~16 wt.%) of water from the feed through the PDMS and PVA membranes respectively. The Arrhenius plot gives an activation energy of 2.49 ± 0.02 kcal mol^{-1} and 2.82 ± 0.02 kcal mol^{-1} for the 2-butanone and water-rich microemulsions respectively.

1. Introduction

Microemulsions are thermodynamically stable, isotropic and microstructured fluid phases containing substantial amounts of oil and water (or brine), surfactant and sometimes co-surfactant. An increasing attention has been paid to these phases because of their unusual physicochemical properties and some potentially large-scale applications of commercial importance such as: detergency, enhanced oil recovery, phase-transfer catalysis, photochemical and polymerization reactions [1–7]. However, only in the last two decades, microemulsions have been used in industrial scale applications. For example; their use as solvents in organic synthesis is steadily rising [8–15]. Microemulsions have also been used to overcome the pH and temperature effects of some pharmaceuticals during their storage [16]. Recently, microemulsion has been used for the homogeneous catalytic oxidation of carbonyl and olefinic groups [17]. However, the isolation of the desired products from the microemulsion mixture has proved to be a difficult task which limits their use. The recovery of the product requires an efficient collapse of the microemulsion. A number of techniques have been tried, for example; the addition of electrolytes followed by ultrafiltration was tried by Yang [18], but the results were not promising. One problem of ultrafiltration is fouling of the membrane by the surfactant.

Despite of the advent in technology, industries are still polluting the environment. For that reason the governments have been ringing the alarm (UN Conference on Human Environment, Stockholm, 1972; UN Conference on the Environment and Development—Earth Summit, Rio de Janeiro, 1992; World Summit on Sustainable Development, Johannesburg, 2002). Nevertheless, research has been expanded to design new techniques to drain off the waste efficiently through they membranes by the surfactants.

Recently, our research has been successful in breaking down two microemulsion systems: cyclohexane/water/*n*-butanol/sodium dodecylsulfate (SDS) and toluene/water/*n*-butanol/sodium dodecylsulfate (SDS) using the pervaporation technique [19,20].

By its large utilization in chemistry and petrochemistry industries, the 2-butanone is considered as an excellent solvent and a good reactant. It can represent also a model for the ketones. In contrast to cyclohexane and toluene studied in our previous work, 2-butanone is relatively more polar. In this case, a microemulsion based on 2-butanone and water can be potentially interesting and worth thinking about. However, the isolation of the 2-butanone from the microemulsion mixture has proved to be a difficult task.

In this work, by using the same extraction method it has attempted to break down two, 2-butanone/water/*n*-butanol/SDS microemulsion systems, one was 2-butanone-rich and the other was water-rich using a hydrophobic-membrane poly(dimethylsiloxane), PDMS and a hydrophilic membrane poly(vinyl alcohol), PVA. In that way, the study has focused on finding the effect of time and temperature on the results. For localizing the break point of these microemulsions with an

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acceptable precision, the study of stirrer rate impact on the pervaporation performance has been carried out.

2. Experimental procedures

2.1. Chemicals

2-butanone and *n*-butanol in 99.8% purity were purchased from Aldrich and distilled in the presence of $CaCl_2$ before use. Sodium dodecylsulfate, supplied by Henkel (Germany), was recrystallized from absolute ethanol. The two-component system, Rhodorsil (silicone RTV-2, PDMS) and its crosslinker agent (RTV-1, hydroxyl-type PDMS derived from a cyclic tetrahydrosiloxane), were supplied by Rhodia (ex-Rhone-Poulenc). PVA provided by Aldrich was used without further purification and had respectively the following characteristics: PVA: ester value (156); hydrolysis degree (88% mol); residual acetyl groups (12%); $\overline{M}_W = 10^6$.

2.2. Microemulsions preparation and characterization

Two microemulsion systems μ_1 and μ_2 were prepared following the procedure described by Bourayne et al. [14]. The microemulsion compositions are shown in Table 1.

The nature of microemulsion was confirmed as follows:

Usually, the dye methylene blue, was used to ascertain the type of microemulsion. Upon adding this dye to the four microemulsions system, an intense blue color was observed.

A filter paper was impregnated with an aqueous solution of cobalt chloride and dried in an oven for about 1 h at 60 °C. On drying, the dyed filter paper turned blue. This blue color became pink in the presence of water. The spot created by a drop of organic compound/ water microemulsion on this dyed paper was surrounded by a pink ring whereas with water/ organic compound microemulsion, no color change was observed.

2.3. Membranes preparation

The PDMS as hydrophobic membrane was prepared as follows: an amount of a mixture of crosslinking agent-PDMS of 5 wt.% was thoroughly and evenly spread over a Teflon-plate surface. The surface was then heated at 50 °C until the film became transparent.

The PVA as a hydrophilic membrane was prepared as follows: a solution containing 10 g wt.% of PVA in water acidified with 4% oxalic acid and 3 drops of sulfuric acid as a crosslinking agent was prepared. This mixture was smoothly spread over a Teflon-plate surface. The plate was then heated with a microwave source using a domestic microwave (Samsung type, 800 W, Korea) for 20 min under nitrogen.

The crosslinked films, PDMS or PVA prepared were swelled in 2-butanone or water respectively and washed several times to extract the residual crosslinking agent, then dried under vacuum at 60 °C for 24 h. The crosslinked-PVA was transparent and became insoluble in water. The thickness of each membrane was about 30 μm.

The crosslinking degrees of PDMS and PVA membranes were respectively 2.5% and 2.3% as determined by the weight difference of each membrane before and after the crosslinking operation. In this work, the choice of this crosslinking degree was based on the optimum of the pervaporation parameters obtained in our previous investigations using cyclohexane/water/SDS/*n*-butanol microemulsion system [19,20]. For reaching this objective, several membranes of PVA were prepared to obtain approximately the same crosslinking degree compared with the PDMS.

Table 1
Microemulsion compositions.

Microemulsion system	2-butanone g (wt.%)	Water	*n*-butanol g (wt.%)	SDS g (wt.%)
μ_1	42.77 (69.5)	6.15 (10)	8.62 (14)	4 (6.5)
μ_2	15.38 (25)	25.50 (54.2)	8.62 (14)	4 (6.5)

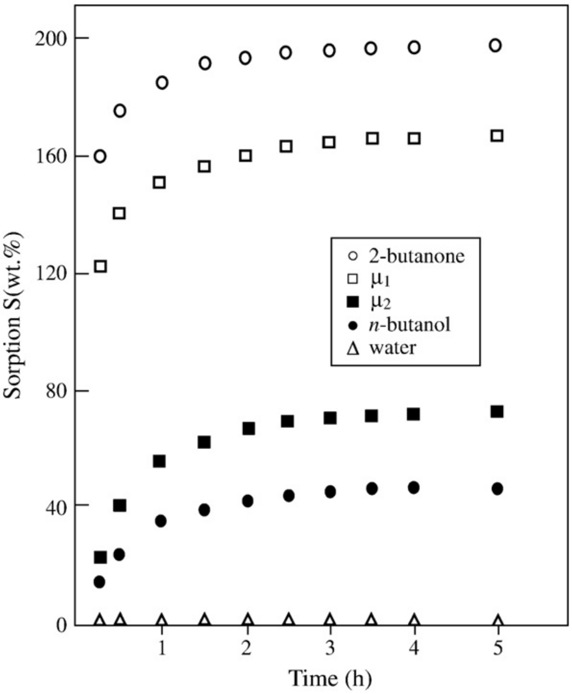

Fig. 1. Plot of the variation of the swelling extent, *S*(wt.%), of PDMS membrane with water, 2-butanone, *n-butanol*, and the formulated microemulsions. Working temperature was 30 °C.

2.4. Swelling measurements and sorption experiments

The dried membrane of about 2.5% crosslinking degree with a known weight was immersed into the microemulsion or its constituents at 30 °C for 5 h. The degree of swelling of the membrane in different liquids during the sorption experiments is given by:

$$S(\%) = \left[\frac{(w - w_0)}{w_0}\right] \times 100$$

where w_0 and w are the sample weights before and after swelling, respectively. The results are plotted in Figs. 1 and 2.

2.5. Pervaporation technique

The pervaporation apparatus used in this work is similar to that reported by Ping et al. [21]. It comprises the following parts: stainless steel pervaporation cell; pervaporation Pyrex-made receiving set fitted with vapor traps; primary vacuum pump (10^{-1} mm Hg); Trivac type.

Different runs were carried out at different temperatures (30–60 °C) and with different stirring rates. The feed was in contact with the membrane for 10 h to reach a steady state before starting the experiment. Different runs were made at least in duplicate and the results were quite reproducible.

The concentration of *n*-butanone, water and *n*-butanol in permeate and retentate was analyzed by gas chromatography using GC-8A, Shimadzu (Japan) with the following parts: TCD detector (katarometer), N_2 as a carrier gas, Cabowax 20 M as a stationary phase, column (2 m in length, 3.17 mm in diameter); injection–port temperature, 180 °C; column temperature, 130 °C, detector temperature, 180 °C.