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Hydrotropic Separation of Mixtures of *o*-/*p*-Hydroxyacetophenones

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ABSTRACT

A new extractive separation technique has been developed for the separation of *o*-/*p*-hydroxyacetophenones (HAPs) using hydrotropy. Hydrotropes are freely water-soluble organic salts which enhance solubility of otherwise water-insoluble or sparingly soluble organic compounds in aqueous solutions. The ability of hydrotropes to differentiate even isomeric organic compounds is explored in this extractive separation. *o*-/*p*-HAPs were extracted from their solutions in organic solvents of different polarities using aqueous solutions of hydrotropes. The solvent nature has a significant effect on the selective extraction of both phenols. The combination of heptane and aq. Na–*p*-toluene sulfonate solution gave almost pure *p*-HAP in the aqueous phase, whereas with chloroform as

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the solvent, it was possible to extract with complete selectivity *o*-HAP into the aqueous hydrotrope solutions.

Key Words: Hydrotrope; *o*-/*p*-Hydroxyacetophenones; Extractive separations; Hydrotropy; Polarity; Aggregation.

INTRODUCTION

Separation of closely related isomeric compounds has always been a challenging problem in the chemical and pharmaceutical industries. Mixtures of *o*-/*p*-hydroxyacetophenones (HAPs) are obtained by acylation of phenol followed by Fries rearrangement and are usually separated by steam distillation as *o*-HAP is steam volatile.[1] However, the ratio of phenol to water in the distillate is low requiring a large amount of steam per kilogram of the phenol for the separation making it energy intensive. Again, the steam condensate from such a process carries a good amount of the dissolved phenol giving rise to the problem of its disposal/recovery. The other methods of the separation of HAPs exploit the difference in their solubility values in non-polar organic solvents.[2] *p*-HAP is less soluble than *o*-HAP in organic solvents and can be separated by solvent crystallization. Although it is possible to get pure *p*-HAP by completely dissolving *o*-HAP in organic solvents, it is necessary to remove the *p*-HAP impurities from these solutions.

Since hydrotropes can distinguish between different organic compounds,[3] as a part of our on-going efforts in the development of newer aqueous solutions based extraction process technology for isomeric/non-isomeric and difficult-to-separate mixtures, we explore here the separation of *o*-/*p*-HAPs using aqueous hydrotrope solutions. Hydrotropes are highly water–soluble organic salts, which when present at sufficiently high concentrations in water, induce increased solubility of other sparingly water-soluble or water-insoluble organic substances. Hydrotropes are structurally amphiphilic having a hydrophilic group and a hydrophobic group which is much smaller in size as compared with that of a conventional surfactant. In a typical hydrotrope, the hydrophobic chain is short, i.e., up to C_6 carbon atom, whereas in the case of surfactants their properties are more prominent with a chain length of C_8 and longer. Hydrotropy, the phenomenon of increasing solubility in the presence of hydrotropes, is considered to be a collective molecular phenomenon possibly occurring by intercalation or co-aggregation of the solute with the hydrotrope molecules. The self-association of the hydrotrope molecules is considered pre-requisite for the solubilization of other organic compounds. The non-covalent self-assemblies of hydrotrope molecules are formed beyond a minimum hydrotrope concentration (MHC) in a manner

analogous to micelles of surface active compounds.[4,5] The solubilization capacity of a hydrotrope depends on the nature of its hydrophobic part, i.e., the alkyl group, as well as on its structure.

The solubility values of *o*-/*p*-HAPs in aqueous solutions of hydrotropes have been recently reported.[6] Although these isomers show different solubilities, the difference is not large enough to separate them by selective solubilization. Aqueous hydrotrope solutions have also been reported to be excellent extractants in solvent extraction or extractive distillation of close boiling point substances.[7–10] The use of hydrotropes in these applications is particularly attractive because of easy product recovery, high selectivity and their higher thermal stability. At the same time, the problem of emulsification, which is normally associated with surfactant solutions, does not occur with the aqueous hydrotrope solutions.[3]

Since the presence of an organic solvent has a substantial effect on the selectivity of extraction, particularly for retaining certain compounds in the organic solvent, we expect better separation of HAPs by using aqueous hydrotrope solutions in a liquid–liquid extraction process. We have used commonly available alkyl benzene sulfonates, in the following study, to investigate their efficiency in the extractive separation of HAP isomers.

MATERIALS AND EXPERIMENTAL METHODS

Sodium salts of *p*-toluene sulfonate (NaPTS) and cumene sulfonate (NaCS), were purchased from Navdeep Chemicals Ltd., Mumbai, and were used as hydrotropes. *o*-/*p*-HAPs were purchased from HIMEDIA, Mumbai, having a reported purity 98%. S.d. Fine Chemicals, Mumbai, supplied heptane, toluene, and chloroform. *o*-HAP is a liquid at room temperature of 30°C (b.p. 213°C/717 mmHg), while *p*-HAP is a solid (m.p. 109°C).

Standard solutions of known concentrations of *o*-HAP (or *p*-HAP) were prepared in different solvents and used as stock solutions. A mixture of an aqueous solution of a hydrotrope and equal volume of organic solution of one of the HAPs was stirred using a magnetic stirrer in a constant temperature water bath at 30°C. After sufficient time was given for equilibration, the organic phase was separated from the aqueous phase by decantation and analyzed on Chemito 8510 gas–liquid dual FID chromatograph (Chemito Technologies Ltd., Mumbai) using a 2.0 m SE-30 stainless steel column with 10% Carbowax. Nitrogen was used as the carrier gas and the oven temperature was maintained at 100°C. The separation experiments were conducted in the same manner using the solutions of *o*-/*p*-HAPs mixtures in organic solvents. The organic phase, in these cases, was analyzed by high-performance liquid

chromatography (HPLC) (Knaur, Germany), at 25°C ± 1°C using 5 μ RP-C_{18} column and methanol–water (80 : 20) as the mobile phase at a flow rate of 0.5 cm^3/min.

RESULTS AND DISCUSSION

Single Component Partition Studies

The partition coefficient of a phenol is defined as the ratio of its concentration in the organic phase to its concentration in the aqueous solution at equilibrium. The effect of the nature of organic solvent on the partition coefficient of individual HAPs was studied for both the hydrotropes. The organic solvents were selected to cover the basic range of polarity. Heptane is a non-polar solvent, whereas toluene is polarizable because of π-electrons. Chloroform is a strongly polar chlorinated hydrocarbon solvent. The reported solubility data show that *o*-HAP is soluble to a large extent in all three solvents.[2] However, *p*-HAP is practically insoluble in heptane and sparingly soluble in toluene (~0.03 mol/kg). In chloroform, however, it has a much higher solubility (~0.8 mol/kg). *o*-HAP has an intra-molecular hydrogen bonding tendency and interacts well with most non-polar organic solvents. *p*-HAP, on the other hand, has a strong inter-molecular hydrogen bonding tendency. Heptane or toluene, which can interact only through London's dispersion forces, cannot break-up the specific H-bonding among the *p*-HAP molecules. The poor solvation of *p*-HAP results in its negligible solubility in these solvents. In chloroform, *o*-HAP is completely miscible and *p*-HAP has a reasonably good solubility. Apparently, the interaction of the HAPs with the organic solvents should become a decisive parameter in extractive separations using hydrotropes too.

Figures 1(a) and (b) show the partition coefficient of *o*-HAP between three solvents and aqueous NaPTS and NaCS solutions, respectively, at different hydrotrope concentrations. Increasing the hydrotrope concentration, the partition coefficient of *o*-HAP decreased substantially; the decrease was appreciably more with NaCS. The partition coefficient decreased to a large extent up to 0.5 mol/dm^3 NaPTS concentration, i.e., a substantial amount of *o*-HAP from the organic phase was transferred to the aqueous hydrotropic solutions. At very low hydrotrope concentrations (<0.1 mol/dm^3), with a partition coefficient of about 60–80, the major amount of *o*-HAP resides mostly in the organic phase.

A MHC, the hydrotrope concentration at which the hydrotropic solubilization of organic substrates becomes prominent, is used to characterize a hydrotrope. The reported MHCs of NaPTS and NaCS are 0.35 and

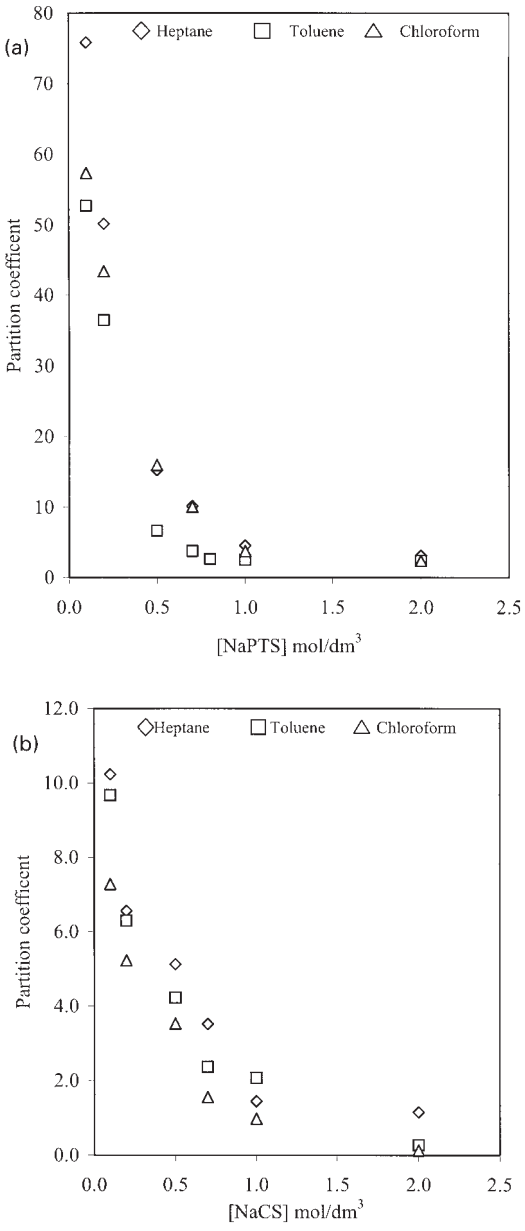

Figure 1. (a) Partition coefficient of *o*-HAP between different solvents and aq. NaPTS solutions. (b) Partition coefficient of *o*-HAP between different solvents and aq. NaCS solutions.

0.1 mol/dm^3, respectively, by surface tensiometry.[4] The partitioning results show a substantial hydrotropic effect on extraction of the HAPs into aqueous solutions even below the MHC of NaPTS. A large change in the partition coefficient of *o*-HAP, below the MHC of the hydrotrope, indicates its probable pre-MHC aggregation, if that is presumably responsible for the transfer of HAPs towards the hydrotropic phase.[4] The solute may also induce an early aggregation of hydrotrope molecules below its MHC. The extraction of an organic solute such as *o*-HAP into aqueous hydrotropic solution may, therefore, be a result of co-aggregation of the solute with the hydrotrope molecules which starts at a much lower concentration of the hydrotrope and would depend upon the nature of the solute and its concentration.

Figures 2(a) and (b) show partitioning of *p*-HAP between toluene and chloroform and aqueous NaPTS and NaCS solutions, respectively. *p*-HAP is sparingly soluble in toluene but has a much higher solubility (0.11–0.59 mol/dm^3) in aq. NaPTS solutions in the concentration range 0.5–2.0 mol/dm^3.[6] These solubility values, themselves, indicate poor partitioning of the phenol towards toluene in the presence of hydrotrope in the aqueous solutions. Indeed the partition coefficient of *p*-HAP is much less than unity and most of *p*-HAP was extracted from the organic phase into the hydrotropic aqueous phase in a single stage extraction. In chloroform, however, the partition coefficient of *p*-HAP is higher than unity.

The decreasing trend of the partition coefficient of *p*-HAP with the hydrotrope concentration remained the same with both the hydrotropes and both the solvents. But the decrease in the partition coefficient of *p*-HAP was much less than that of *o*-HAP. The individual partitioning of both HAPs, indicated a substantial and differential effect of the hydrotropes in the aqueous solutions. For both the isomers, the partition coefficient asymptotically reached to constant values at higher hydrotrope concentrations.

Aromatic sulfonate hydrotropes, being amphiphilic, show a self-aggregating tendency which is particularly strong if the alkyl chain of the hydrotrope is sufficiently long. The self-association of NaPTS molecules, because of the presence of only methyl group on the aromatic ring is debatable. However, the experimental evidence points towards lower polarity and higher microviscosity experienced by the dissolved organic probes in the aqueous hydrotrope solutions indicating their microenvironment having properties different from those of bulk water.[4] The self-aggregation of hydrotropes is presumably responsible for the increased solubilization of organic solutes in the aqueous solutions of hydrotropes.[4] The main point of debate, however, is about the shape and structures of the aggregates of hydrotropes. Srinivas et al.[5] have studied the crystal structures of the aromatic sulfonates in solid phase which show distinct hydrocarbon and ionic regions which alternate in the solid crystals. In the presence of water, however, it is difficult to

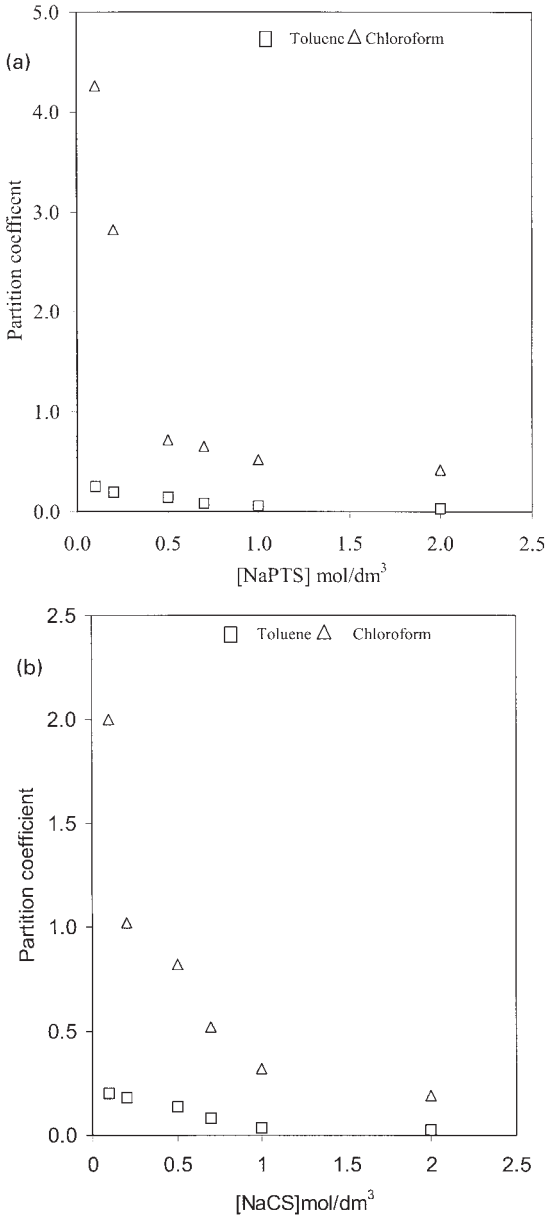

Figure 2. (a) Partition coefficient of *p*-HAP between different solvents and aq. NaPTS solutions. (b) Partition coefficient of *p*-HAP in different solvents and aq. NaCS solutions.

envisage such long-range structures because of ionic nature of headgroups of the hydrotropes which get strongly solvated by water. The electrostatic repulsion between the headgroups would not permit close packing of the hydrotropes keeping their aggregation numbers low in their self-assemblies. Recent small-angle neutron scattering studies indicate near spheroidal aggregation of a closely related hydrotrope sodium *n*-butyl benzene sulfonate with aggregation number close to 30.[11] NaCS can be assumed to have similar self-associated structures in water. The alkyl chains of the hydrotropes would decide the way these molecules can self-assemble in the aqueous solutions. Irrespective of the shape and size of the aggregates it is clear that self-organization of hydrotropes, either alone or with the dissolved solutes, will be responsible for increased solubilization and in the present case for the increased extraction of HAPs into the aqueous hydrotrope solutions. The decreasing partitioning of an organic solute towards the organic phase and its increased concentration in the hydrotrope solutions, indicate that the solute must be someway associated with the hydrotrope aggregates. Because of the lack of any hydrocarbon core in the hydrotrope assemblies, unlike that in micelles of conventional surfactants, the solute is envisioned to co-aggregate with the hydrotrope by intercalation between two hydrotrope molecules in the assembly. A molecule, which fits well in such a manner, should be preferred by the hydrotrope aggregates. While any other molecule having difficulty in fitting itself in the hydrotrope assembly gets rejected giving the selectivity in solubilization and as well as in the extraction. In the presence of an organic solvent, the relative ease by which the solute is solvated either by the organic solvent or by the non-polar part of the hydrotrope assemblies in aqueous solutions should decide the efficiency of the separation and particularly the selectivity of extraction. From the above results, it is apparent that the relative affinities of solvent and the hydrotrope solutions will decide the final separation efficiency of the hydrotropic extraction process.

Binary Partitioning Studies

Although *p*-HAP is practically insoluble in heptane, in the presence of *o*-HAP it has a very limited solubility owing to strong solute–solute interactions amongst the isomers.[2] An 80 : 20 mixture of *o*-/*p*-HAPs was investigated for the separation. When this mixture is added to heptane, *p*-HAP precipitates in almost pure crystalline form and the resulting organic solution contains all *o*-HAP of the initial mixture and a small amount of *p*-HAP. On contacting this organic solution with an equal volume of the aqueous NaPTS solution, *p*-HAP was preferentially (85–90%) extracted from the

organic phase, whereas less than 10% of *o*-HAP was extracted into the hydrotropic phase (Fig. 3). With the increase in hydrotrope concentration, the extraction of both components marginally increased. After a two-stage cross-flow extraction, the raffinate organic phase contained almost pure *o*-HAP with negligible traces of *p*-HAP. Figure 4 shows the separation factor of *o*-/*p*-HAPs using NaPTS as the hydrotrope for extraction from

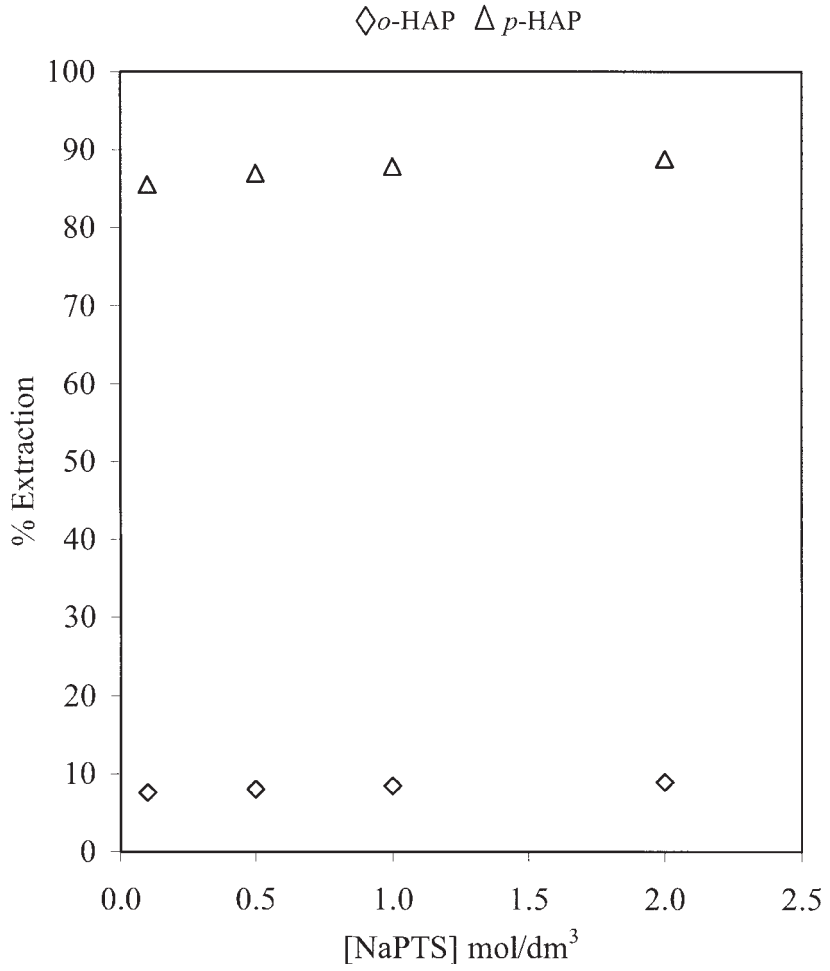

Figure 3. Percentage extraction of *o*-HAP and *P*-HAP using aq. NaPTS solutions. Solvent: heptane; initial total concentration of phenols: 1.0 mol/dm^3.

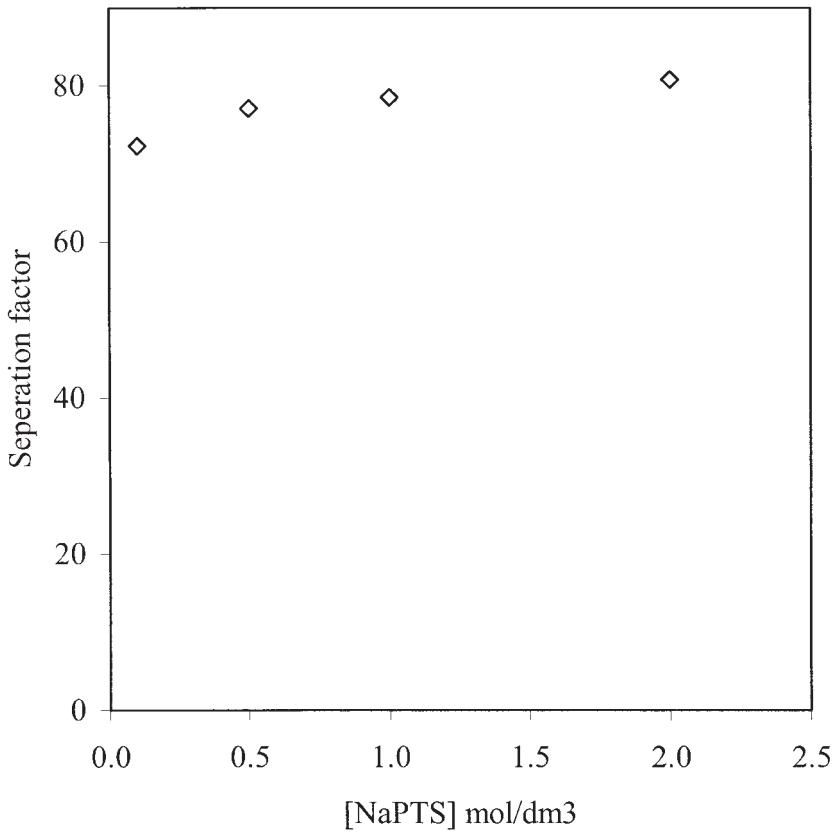

Figure 4. Separation factor of *o*-HAP and *p*-HAP mixtures by extraction using aq. NaPTS solutions. Solvent: heptane; initial total concentration of phenols: 1.0 mol/dm^3.

their heptane solutions. Separation factor for the single stage of extraction is defined by Eq. (1)

$$\alpha = \frac{[p - \text{HAP}/o - \text{HAP}]_{\text{aq}}}{[p - \text{HAP}/o - \text{HAP}]_{\text{org}}} \tag{1}$$

The values of separation factor, as high as 80, were obtained in the heptane–NaPTS system for a single stage contact. The selectivity was slightly better at higher hydrotrope concentrations.

Although toluene solubilizes *p*-HAP to a small extent, in the presence of *o*-HAP its solubility in toluene increased to 0.1 mol/dm^3. Figure 5 shows the extraction of *o*-HAP and *p*-HAP from toluene using aqueous NaPTS solutions.

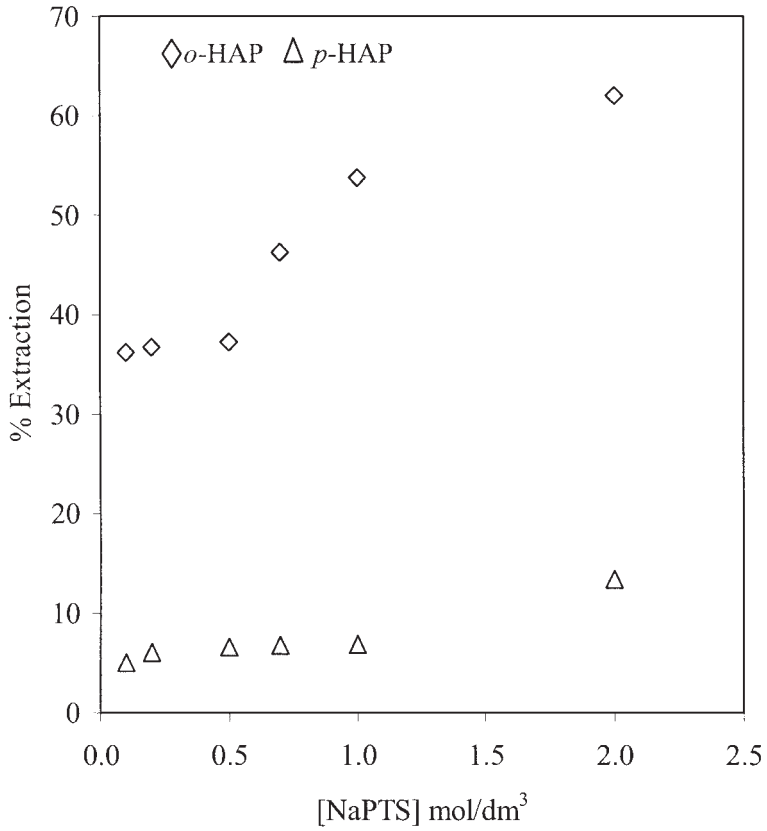

Figure 5. Percentage extraction of *o*-/*p*-HAPs (90 : 10) mixtures using aq. NaPTS solutions. Solvent: toluene; initial total concentration of phenols: 1.0 mol/dm^3.

Surprisingly, there was a reversal of the selectivity of the extraction from that obtained for extraction from heptane. Unlike the extraction from heptane, the extraction of *o*-HAP was preferential. Using 2.0 mol/dm^3 of NaPTS solution, ~62% of *o*-HAP was extracted, whereas only ~19 % of *p*-HAP was extracted from the toluene phase in a single stage extraction. At lower NaPTS concentrations, the % extraction of *p*-HAP was just 5% which remained more or less constant upto 1.0 mol/dm^3 hydrotrope concentration, whereas that of *o*-HAP suddenly increased at 0.5 mol/dm^3 hydrotrope concentration.

It is obvious that the nature of the solvent and its interaction with the phenol(s) have a substantial effect on the extraction and separation efficiencies of the hydrotropic extraction process. The change in the feed composition did

not have any significant effect on the percentage extraction of HAPs, at least in the range investigated which was mainly rich in *o*-isomer. Also, a relatively larger effect on percentage extraction of *o*-HAP when compared with that of *p*-HAP, after a certain concentration of the hydrotrope, indicates competitive solubilization of *o*-HAP within the hydrotrope assemblies. The structure of *o*-HAP, with both substituent groups on one side of the aromatic ring, makes it somewhat amphiphilic, whereas, in the case of *p*-HAP the two groups, both having more electronegative oxygen, are at the opposite ends of aromatic ring. It is possible that *o*-HAP fits well into the hydrotrope assemblies having distinct hydrophobic and hydrophilic regions. On the other hand, the association of *p*-HAP with the amphiphilic molecules seems energetically unfavorable and it prefers the toluene phase. Although, in the case of heptane as solvent, *p*-HAP showed the preferential partitioning towards the aqueous hydrotrope solutions, it is driven more by poor solvation by heptane than by its affinity towards the hydrotrope aggregates in the aqueous phase. It seems that the hydrotrope molecules adjust themselves around the solute depending upon the nature of the solute.

Figures 6 and 7 demonstrate percentage extraction of *o*- and *p*-HAPs from chloroform using aqueous NaPTS solutions for 80 : 20 and 90 : 10 mixtures of both isomers. There was an increase in the percentage extraction of *o*-HAP with increasing hydrotrope concentration, whereas a decreasing trend was obtained for the percentage extraction *p*-HAP unlike the results with heptane and toluene. These results confirm that the solvent nature does play an important role in the extraction efficiency and selectivity, while using hydrotrope solutions for extraction. The competition between *o*-HAP and *p*-HAP also was observed such that at the low concentrations of the hydrotrope, there was poor extraction of *o*-HAP and both the isomers were extracted into the hydrotropic phase. As the hydrotrope concentration was increased, the extraction of *p*-HAP decreased and that of *o*-HAP increased. At 2.0 mol/dm^3 NaPTS concentration, *o*-HAP was extracted so preferentially that *p*-HAP was completely retained in the organic phase. The effect of the nature of the solvent in the case of *p*-HAP has to be considered to explain its poor extraction into the hydrotrope solutions. The strong solute–solvent interactions retain *p*-HAP in the organic phase. The separation studies were also conducted using NaCS as the hydrotrope, the results of which are shown in Fig. 8. With NaCS, the trend in the percentage extraction was the same as that with NaPTS. However, at 2.0 mol/dm^3 of NaCS concentration there was still an appreciable extraction of *p*-HAP into the aqueous hydrotropic phase. NaCS is hydrophobically a bigger hydrotrope than NaPTS because of the presence of isopropyl group on the aromatic ring and is expected to show better solubilization of organic compounds than NaPTS.

The partition experiments were conducted with different volume ratios of the aqueous hydrotrope phase to organic chloroform phase. The effect of

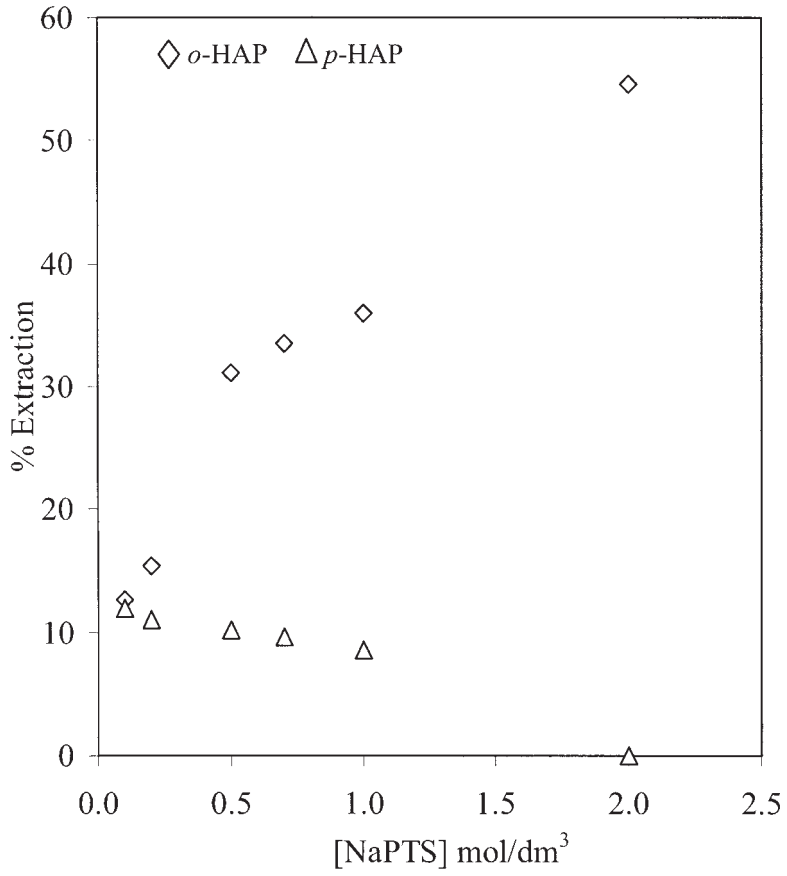

Figure 6. Percentage extraction of *o*-/*p*-HAPs (80 : 20) mixtures using aq. NaPTS solutions. Solvent: chloroform; initial total concentration of phenols: 1 mol/dm^3.

volume change on the percentage extraction is shown in Fig. 9. At the lower volume ratios, the extraction of *p*-HAP was expectedly still lower. At 0.5volume ratio, the percentage extraction of *o*-HAP was 30 %, but as the ratio was increased, the percentage extraction of *o*-HAP increased but did not affect *p*-HAP at all. With the volume of the aqueous phase, twice as much as the organic phase, 72% extraction of *o*-HAP could be achieved in a single stage. Increasing the aqueous phase volume, increases the number of hydrotrope aggregates exposed to the fixed amount of the solute in the organic phase. Hence, the capacity of the hydrotrope solution to extract HAPs from the organic phase also increased. But interestingly negligible

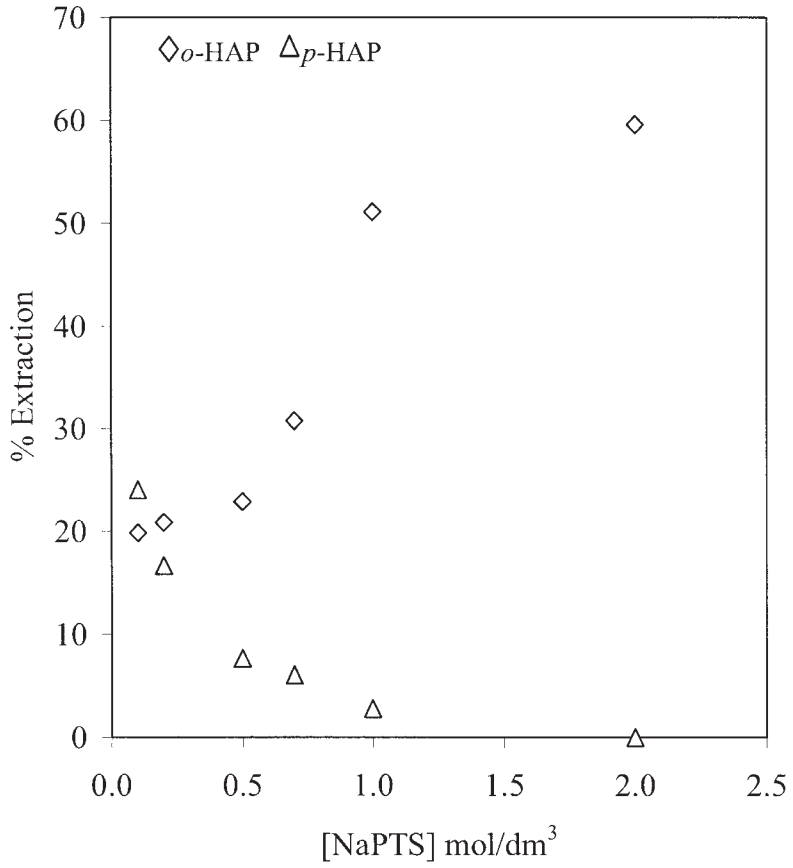

Figure 7. Percentage extraction of *o*-/*p*-HAPs (90 : 10) mixtures using aq. NaPTS solutions. Solvent: chloroform; initial total concentration of phenols: 1 mol/dm^3.

extraction of *p*-HAP was observed into the aqueous phase giving a much higher selectivity towards *o*-HAP.

The organic solutes from hydrotropic phase are usually recovered by dilution with water. But this method requires a large amount of water to bring hydrotrope concentration below its MHC. The recycle of hydrotrope solution for primary extraction requires concentration of the diluted hydrotrope solutions by evaporating water. This makes the overall process energy intensive and probably uneconomical. Considering the extraction results with solvents of different polarities, it must be possible to recover the dissolved solute from the hydrotrope solutions by a secondary solvent extraction using a

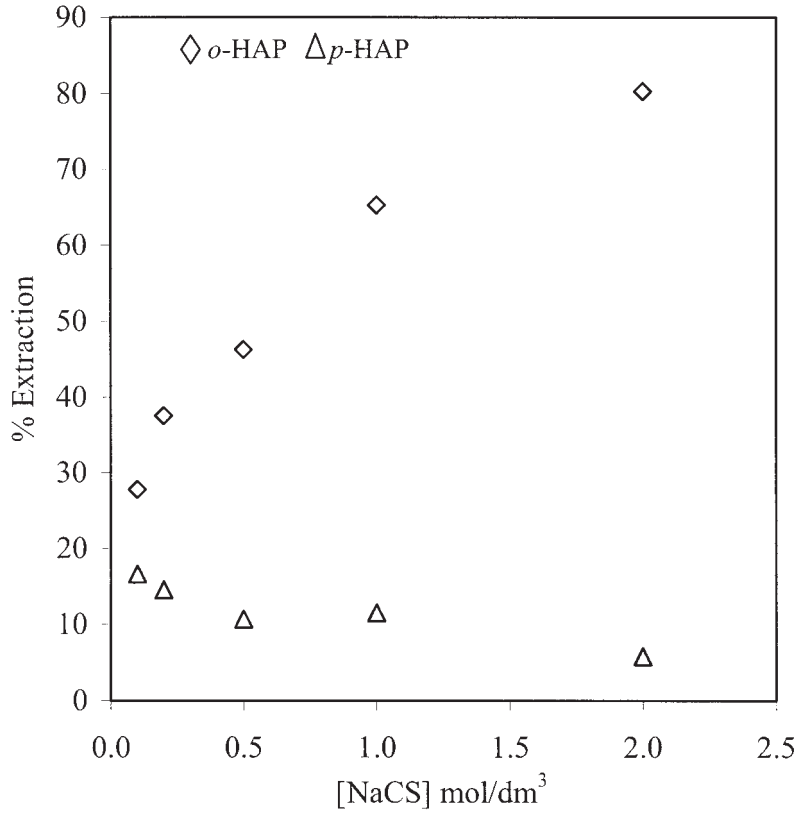

Figure 8. Percentage extraction of *o*-/*p*-HAPs (90 : 10) mixtures using aq. NaCS solutions. Solvent: chloroform; initial concentration of phenols: 1 mol/dm^3.

solvent of appropriate polarity. Using ethylene dichloride and chloroform in equal volume ratio, the amount of *o*-HAP extracted from the hydrotropic phase (2.0 mol/dm^3 NaPTS) was 14% and 48%. Toluene is, therefore, a better solvent for the recovery of *o*-HAP after the primary hydrotropic extractive separation. Although the extraction using aqueous hydrotrope solutions can separate *o*-/*p*-HAPs, the single component partitioning data could not be extended directly for the estimation of separation efficiency. The hydrotrope aggregates thus either have a limited capacity of solubilization or structural changes in the hydrotrope assemblies take place in the presence of the dissolved solute(s). Dissolution of one solute molecule in hydrotrope assemblies may exclude the second molecule which, however, cannot be generalized to other systems.

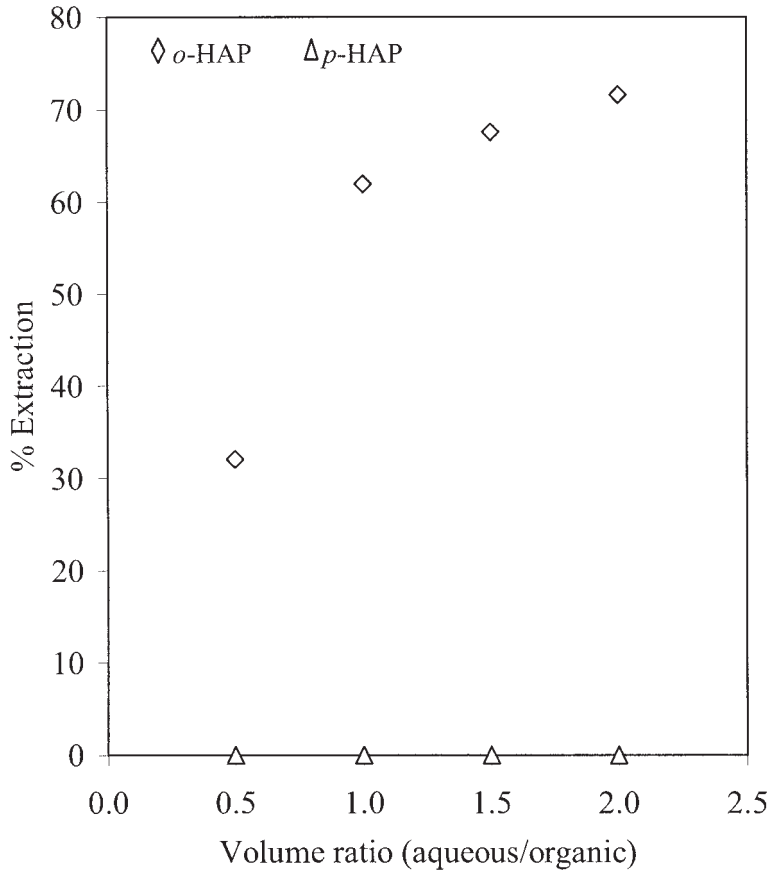

Figure 9. Comparison of extraction of *o*-HAP and *P*-HAP with respect to volume ratio. Hydrotrope: 2 mol/dm^3 NaPTS; solvent: chloroform.

CONCLUSIONS

The hydrotrope solutions can distinguish between *o*-/*p*-isomers of HAP. The extractive separation process gives a good selectivity for the *o*-/*p*-HAPs mixtures, if combined with selective solubilization of *o*-HAP in the inert organic solvents. The heptane–aqueous NaPTS system is effective in preferential extraction of *p*-HAP from heptane. Pure *o*-HAP can be recovered then from the organic phase. With the chloroform–NaPTS system, pure *o*-HAP can be extracted into aqueous hydrotropic phase while retaining *p*-HAP in the organic solutions.

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