

Phase equilibrium in the systems (alkanes, or alcohols, or ionic liquids with fragrance raw materials) (A review)

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Introduction

Fragrance materials are used mainly in food and cosmetic industry to give scent to a number of products. Fragrances date back to the ancient civilisations – it is known that Egyptians used perfumes, including vegetable oil, incenses and mixtures of resins. The knowledge of phase equilibria is required to design processes for obtaining new fragrance materials. The Japanese studies showed that 3-methylbutanol, heptanal and 2-undecanone are substances responsible for giving a certain fish-like odour to food products [1]. Another studies showed that 2-heptanone is a hydrophobic pheromone that has a negative impact on bees collecting nectar from flowers [2]. If it is present in high concentrations, it serves as a repellent and disturbs the nectar collecting process, whereas, in case of low concentrations, it can exhibit luring properties [3–7]. Binary systems of 1-alcohols with ethylene glycol monomethyl ether are used in the chemical industry as solvents for oils and petrol to increase their octane number [8]. Toxicological properties of ethyl acetate, a well-known fragrance, and its application in such products as toilet soap, shower gel, face cream and body lotion can be found in the paper [9]. The analysis of amylcyclohexyl acetate phototoxicity has shown that it does not absorb UV radiation in the range 290–320 nm, thus it can be used in small amounts as a cosmetic additive [10]. 2,3-Pentanodione is a popular ketone used in the production of beer and wine [11, 12]. Diketones are biodegradable solvents commonly used as inhibitors in the polymerisation reaction and as substrates in the pharmaceutical industry. 2-Phenylethanol is an important and very expensive substance usually used in food, cosmetics and perfume industry for production of beverages, candies, ice-creams, puddings, chewing gums, cookies etc. [13–15].

The Research Institute for Fragrance Materials tests various fragrance materials and collects available data related to them. Hundreds of substances, acids, esters and alcohols are considered to be key additives to food, pharmaceuticals and cosmetics [16, 17]. There are known lists of perfumes, fragrance materials, chemical additives along with their toxicity assessments [18–20].

In recent years, the laboratory of the Department of Physical Chemistry, Warsaw Technical University, has studied phase equilibria of compounds such as long-chain alcohols (C_8 , C_{10} , C_{12}), ketones like 2-heptanone, 2-nonanone [21], cyclohexane derivatives such as cyclohexyl acetate, cyclohexylacetic acid [22] and finally 2-phenylethanol (PEA) [23–25]. The solubility of these compounds in water, alcohols, long-chain hydrocarbons, ionic liquids (ILs) and eutectic mixtures of type (choline + oxalic acid) was also studied [25]. This involved the use of ILs with various cations such as imidazolium, pyridinium, isoquinolinium, pyrrolidinium, piperidium and ammonium and anions such as bis(trifluoromethylsulfonyl)imide ($[NTf_2^-]$), thiocyanate ($[SCN^-]$), tosylate ($[TOS^-]$), tetracyanoborate

($[TCB^-]$), tricyanomethanide ($[TCM^-]$), dicyanamide ($[DCA^-]$) and others. Solid-liquid and liquid-liquid phase equilibria were studied in binary systems, as well as liquid-liquid equilibria in ternary systems (IL + PEA + water). The solubility of these compounds depending on the anion and the alkyl substituent in cation depends on their melting point and melting enthalpy, as well as intermolecular interactions in the solution. Simple eutectic mixtures fully miscible or with a miscibility gap with upper critical solution temperature were found in binary systems with organic solvents. However, it was observed that quinolinium- and ammonium-based ionic liquids show an increased selectivity of PEA extraction from water [26, 27].

Ketones, alcohols, cyclohexane derivatives

Phase equilibria were studied in binary systems of fragrance materials. Three long-chain alcohols: 1-octanol, 1-decanol, 1-dodecanol as well as five long-chain ketones: 2-heptanone, 2-nonanone, 2-undecanone, 2-dodecanone i 2-tridecanone were analysed [21]. Suitable mixtures of these compounds create floral, fruity and herbal fragrances. Solid-liquid equilibrium measurements were performed by means of the dynamic method by observing the temperature of the last crystal disappearance. Simple eutectic systems with full miscibility in the liquid phase were found. It was observed that the solubility of ketones in given alcohol decreases with the an increase in the carbon chain length from heptanone to tridecanone, which is related to their melting point. The eutectic point shifts towards a higher mole fraction of ketone as the carbon chain length of alcohol increases. The eutectic point shifts towards the solution of high alcohol concentration as the carbon chain length of ketone increases; eutectic temperature an increase with the an increase in the ketone chain length. Eutectic point temperature increases with the increase in the alcohol carbon chain length.

Moreover, phase equilibria of 2,3-pentanodion in binary systems were measured, including those with alkanes (tridecane, octadecane, eicosane) with cyclohexane and alcohols (1-hexadecanol, 1-octadecanol, 1-eicosanol [28]). Simple eutectic systems were found in alcohol systems and the occurrence of miscibility gap in the liquid phase was found in alkane systems (Fig. 1). All presented systems had an upper critical solution temperature (UCST).

Cyclohexane derivatives formed a large group of tested compounds. It included cyclohexylcarboxylic acid (CCA), cyclohexylacetic acid (CAA), cyclohexyl acetate (CA), 2-cyclohexylethyl acetate (2CEA) and 2-cyclohexylethanol (2CE). A natural CCA can be found in coffee, and as a fragrance or flavour, or as both: in blueberries, cherries, tropical fruits, pineapples, raisins and raspberries. CAA is used as a fragrance or flavour, or as both in caramel, cocoa, chocolate, coffee, honey and maple syrup. Natural CA is present in cabbage and is used as a fragrance or flavour in apples, bananas, black currant and rum. 2CEA is used as a fragrance or flavour for the production of floral, raspberry and rose fragrances. 2CE is the most known material which is used as a fragrance or flavour in over 100 different materials, among which the most important ones are green apple, cognac, guava, kiwi, magnolia, nut and rose.

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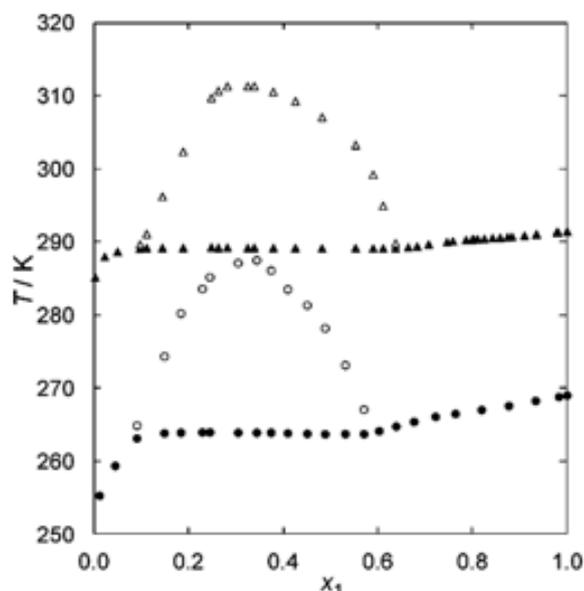


Fig. 1. Phase diagrams for $\{n\text{-alkane (1)} + 2,3\text{-pentanodion (2)}\}$ systems; ●, tridecane SLE and ○, LLE; ▲, hexadecane SLE and △, LLE

Phase equilibria were also measured for binary systems of cyclohexane derivatives and alcohols (1-octanol, 1-decanol, 1-dodecanol) and water, at normal pressure and at increased pressures up to 1000 MPa [22, 29, 30].

Basic fragrance materials exhibit strong interactions with solvents, both with donor and acceptor ones. Alcohols are compounds that form hydrogen bonds. The solubility of CCA and CAA in alcohols depends on the compound melting point, which is similar in this case. The solubility of CAA in given alcohol is slightly better than that of CCA. The results indicate simple eutectic systems with full miscibility in liquid phase. Just as for previous results, CCA and CAA carboxyl group is responsible for strong specific interactions with alcohols. Eutectic temperature of the tested systems increases with the increase of molar mass of 1-alcohols. Replacing $-\text{COOH}$ group with $-\text{CH}_2\text{COOH}$ group in a cyclohexane ring, resulted in the shift of the eutectic point towards higher alcohols. The solubility of other fragrance materials: CA and 2CEA, which are liquids at ambient temperature is similar in 1-alcohols and depends mainly on the melting point of 1-alcohol.

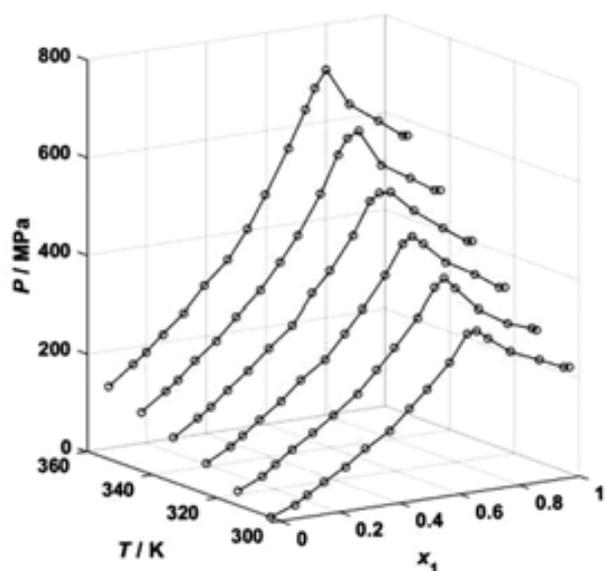


Fig. 2. Eutectic diagram for $\{1\text{-octanol (1)} + \text{CCA (2)}\}$ [31]

In the food technology, there is an increasing interest in processes conducted at high pressures (200–500 MPa). The

greatest advantage of applying high pressure in food technology is the fact that regardless of the size, shape and composition of the product, the pressure “penetrates the entire mass”. High-pressure applications are combined with temperatures to obtain certain food properties: gelling, protein stability, fat migration, microbiological or enzymatic activation. High-pressure systems can be also used for sterilization of food products. An example of the effect of pressure on the tested systems is presented in Figure 2.

A difference in the melting point under atmospheric and increased pressure is much bigger for 1-octanol than for CCA. In case of 1-octanol, the pressure change of 950 MPa resulted in the change of the melting point by 90 K, whereas the pressure change of 150 MPa for CCA caused a temperature change of 50 K [30].

Furthermore, wide-spread studies of water solubility were carried out. The following substances were tested: cyclohexylcarboxylic acid, cyclohexylacetic acid, 2-cyclohexylethanol, cyclohexylethyl acetate and cyclohexyl acetate, 2-heptanone, 2-nonanone, 2-undecanone, 2-dodecanone, 2-tridecanone and long-chain alcohols 1-decanol and 1-dodecanol [22]. The obtained phase diagrams showed equilibria of the liquid-liquid and/or liquid solid type. The obtained results indicated that in case of cyclic fragrance materials, the lowest miscibility gap with water can be observed for 2-cyclohexylethanol, whereas the greatest one for cyclohexyl acetate. In case of ketones, physicochemical properties are affected by both the length of the carbon chain and its evenness. Both systems with fragrance materials of structure derived from cyclohexane and systems with ketones had the upper critical solution temperature (UCST).

2-Phenylethanol

2-Phenylethanol (PEA) is an aromatic alcohol of oil-like consistency with a pleasant rose scent [31, 32]. It can be found in the highest concentration in rose petals, from which it can be obtained by means of extraction. This is one of the most popular fragrances used currently in the industry [33], and the second most popular after vanillin [34]. Thanks to its fragrance properties it is used in food, cosmetic and perfume industry [35]. The studies indicate that PEA has also potential to be a biofuel due to its high energy density in comparison to other alcohol biofuels [36] and its antibacterial properties [37]. The PEA is naturally present as one of the essential oils in many plants such as roses, hyacinth, jasmine, narcissi and lilies [38]. In most cases, PEA content in natural oils is too low, which makes that obtaining it from plants is uneconomical and laborious. Rose oil is an exception containing up to 60% of 2-phenylethanol [39]. PEA derivatives such as certain fat acid esters and ethers, e.g. phenylethyl acetate or propionate also has a strong rose scent, which makes PEA a substrate used for the production of its fragrance derivatives [40]. Globally, over 10,000 t of PEA per year is consumed, approx. 7000 t of which is produced by means of the synthesis [41]. Synthetically obtained PEA is cheap, however it requires the use of toxic substances that are harmful to health in the production process, rendering this method unattractive in terms of the environment protection. Unfortunately, the use of fragrance additives obtained by means of the chemical synthesis in beverages, food products and cosmetics has been legally restricted pursuant to the Act applicable in the USA and in the EU [42]. Obtaining PEA from plants is the least profitable method. There are several techniques for obtaining essential oils from plants, including solvent extraction, cold pressing, supercritical extraction; however, steam distillation is the most popular [43]. A solution may come from biotechnology, starting from the most efficient method of L-phenylalanine bioconversion, the product of which is defined as natural. However, the production involving microorganisms such as yeast is limited by PEA inhibitory effect on the reaction rate. It is necessary to remove the product to increase reaction yield, e.g. by means of the two-phase *in situ* extraction. The extraction involves

the use of alcohols, oleic acid, oleic alcohol, long-chain hydrocarbons and ILs [22, 31 ÷ 37]. For PEA extraction from aqueous solutions using ILs such as [MPPyr][NTf₂], [OMA][NTf₂] and [BMIM][NTf₂], even 3- or 5-fold increase in reaction yield was observed [38]. ILs with [NTf₂]⁻ anion show potential for extraction applications in aqueous environment for PEA production. Previously, ILs with ammonium cation proved to be non-toxic, cheap and environmentally friendly, whereas liquids with [NTf₂]⁻ anion usually show a limited miscibility with water. The studies covered also phase equilibria in binary systems (IL + PEA or water) and in ternary systems (IL + PEA + water). They aimed to evaluate the extraction capacities of ammonium ionic liquids with [NTf₂]⁻ anion [44] and many others ionic liquids [23, 25 ÷ 27].

An effective extractant should have a low-water solubility, high PEA-affinity and show no toxicity towards production strains. ILs are an attractive alternative for traditional solvents. Depending on the chosen anion and cation, such IL properties as viscosity, density, thermal stability and miscibility with other substances can be controlled. This involved the use of ILs with various cations such as imidazolium, pyridinium, isoquinolinium, pyrrolidinium, piperidinium and anions such as bis(trifluoromethylsulfonyl)imide ([NTf₂]⁻), thiocyanate ([SCN]⁻), tosylate ([TOS]⁻), trifluoroacetate ([TFA]⁻), tetracyanoborate ([TCB]⁻), trifluorotris(perfluoroethyl)phosphate ([FAP]⁻), dicyanamide ([N(CN)₂]⁻), tricyanomethanide [C(CN)₃]⁻, 4,5-dicyanamide-2-(trifluoromethyl)imidazolidine and others. [23, 25–27]. In binary systems with water, they fill the miscibility gap [45]. All the tested ionic liquids show full miscibility with PEA. As an example, a liquid-liquid phase equilibrium is presented for the (IL + PEA + water) ternary system using ammonium liquid, bis{(trifluoromethyl)sulfonyl}-N-methyl-N-trioctylammonium imide, [N₁₈₈₈][NTf₂] (Fig. 3).

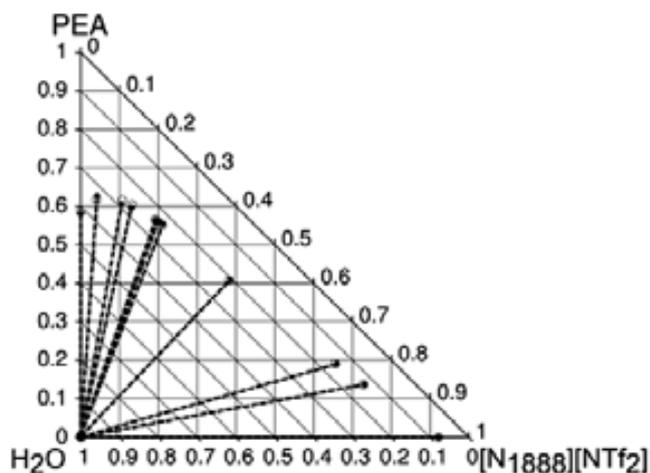


Fig. 3. Plot of the experimental and calculated (liquid + liquid) phase equilibrium of { [N₁₈₈₈][NTf₂] (1) + PEA (2) + water (3) } ternary system at temperature $T = 308.15 \text{ K}$: ●, experimental data; ○ calculated with NRTL equation, parameter $\alpha = 0.2$

To evaluate the choice of the extractor, the selectivity and the partition coefficient are calculated with the following formulas:

$$S = \frac{x_2^{\text{II}} \cdot x_3^{\text{I}}}{x_2^{\text{I}} \cdot x_3^{\text{II}}} \quad (1)$$

$$\beta = \frac{x_2^{\text{II}}}{x_2^{\text{I}}} \quad (2)$$

where: x is a mole fraction. I and II refer to the water-rich or ionic-liquid rich phase. References 2 and 3 correspond to PEA and water, respectively. The obtained average selectivity values (S_{av}) for ammonium liquids are as follows: [N_{1112OH}][NTf₂] ($S_{\text{av}} = 130$) < [N₁₁₁₄][NTf₂] ($S_{\text{av}} = 294$) < [N_{2212OCH₃}][NTf₂] ($S_{\text{av}} = 319$) < [N₂₂₂₈][NTf₂] ($S_{\text{av}} = 711$) <

[N₁₈₈₈][NTf₂] ($S_{\text{av}} = 806$) [44]. For other liquids: [COC₂C₁PYR][NTf₂] ($S_{\text{av}} = 293$) < [C₆DBCO][NTf₂] ($S_{\text{av}} = 347$) < [COC₂C₁IM][NTf₂] ($S_{\text{av}} = 394$) < [C₆C₁PYR][NTf₂] ($S_{\text{av}} = 424$) [27]. A very high selectivity was also found for bis{(trifluoromethyl)sulfonyl}-N-octylisoquinolineimide, N-italic ([C₈iQuin][NTf₂]), ($S_{\text{av}} = 795$) [27]. Average extractant partition coefficients (β_{av}) are within the range from 18 to 350. As an example, for ammonium ionic liquids they are as follows: [N_{1112OH}][NTf₂] ($\beta_{\text{av}} = 72$) < [N₁₁₁₄][NTf₂] ($\beta_{\text{av}} = 108$) < [N_{2212OCH₃}][NTf₂] ($\beta_{\text{av}} = 126$) < [N₁₈₈₈][NTf₂] ($\beta_{\text{av}} = 185$) < [N₂₂₂₈][NTf₂] ($\beta_{\text{av}} = 209$) [44].

Summary and conclusions

Fragrance materials with the structure derived from cyclohexane show a good miscibility with alcohols and they form simple eutectic systems, whereas with water, they have the miscibility gap. The smallest miscibility gap with water is observed for 2-cyclohexanol, which is inherently related to its structure and formation of hydrogen bond with water. A slightly worse solubility was found for cyclohexylacetic acid, which is also due to the interaction of polar carboxyl group with water. It may be observed that introducing -CH₂- group into the molecule of carboxylic acid results in the increased solubility of cyclohexylacetic acid in water. The worst solubility among the compounds with the structure derived from cyclohexane was observed for cyclohexyl acetate and 2-cyclohexylethyl acetate. Similarly, in case of both of these compounds, it can be observed that the introduction of the ethyl group results in the increase in the solubility.

Ketones form systems that have limited miscibility with water. The miscibility gap increases with the an increase in the carbon chain length. Therefore, the biggest miscibility gap is observed for 2-tridecanone and the smallest one – for 2-heptanone. It was observed that the ketone solubility depends on the number of carbon atoms in the molecule and its evenness.

Attempts were made to find a new PEA extractor from aqueous phase for the bioproduction process. A very good selectivity and yield were found for two ammonium ionic liquids and an isoquinolinium ionic liquid. The results reflect the structure and the polarity of solvents and ILs and their mutual interactions. The choice of ILs involves the introduction of polar functional groups into a cation or an anion, thus enabling strong interactions with other molecules in the solution via the hydrogen bond, π - π or n - π type interactions, ionic or charge transfer interactions, or van der Waals interaction, dispersive and non-specific interactions. There are many bonds in the solution at the same time, the most important of which are interactions between the extractor and the extracted substance.

The conducted studies will make it possible to design new compositions of mixtures in a deliberate and more precise manner than it has been done previously, taking into account fragrance materials added to give a certain scent to cosmetics or food products, or – for odour-less products – in order to hide an undesirable odour. Thermodynamic studies on ionic liquids as extractors are still in progress. Better understanding of these properties may provide information required for new technologies. It is important to propose such ILs that have a low density and viscosity and that are cheap, recyclable, non-toxic and non-flammable for process applications.

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Na posiedzeniu Rady PIPC 9 września br. uzupełniono jej skład oraz wybrano Przewodniczącego. Na Przewodniczącego Rady powołano Mariusza Bobera, Prezesa Zarządu Grupy Azoty S.A. Wiceprzewodniczącym Rady został Zbigniew Gagat, Wiceprezes Zarządu Grupy Azoty Zakładów Azotowych Puławy S.A. Tym samym uzupełniono skład Prezydium Rady (w skład Prezydium wchodzi obecnie przedstawiciele pięciu podmiotów członkowskich PIPC. Poza nowo wybranymi, członkami-Wiceprzewodniczącymi, w Prezydium Rady są: Piotr Chelmiński (Członek Zarządu PKN ORLEN S.A.), Ewa Anna Wołynkiewicz (Brenntag Polska Sp. z o.o.) oraz Zbigniew Warmuz (Wiceprezes Synthos S.A.).

Na tym samym posiedzeniu Rady uzupełniono również skład organu. W skład Rady weszli: Mateusz Gramza (Prezes Zarządu Grupy Azoty Zakładów Azotowych Kędzierzyn S.A.), dr Mateusz Aleksander Bonca (Wiceprezes Zarządu Grupy Lotos S.A.), Maciej Kropidłowski (Dyrektor Biura Rozwoju i Technologii w PKN ORLEN S.A.), Tomasz Laudy (Prezes Zarządu Qumak S.A.).

Pracami Polskiej Izby Przemysłu Chemicznego kieruje jednoosobowy Zarząd. Funkcję Prezesa Zarządu pełni dr inż. Tomasz Zieliński. (*abc inf. prasowa PIPC, 9 września 2016 r.*)

Andrzej Stachnik przejmuje stery w Biomed-Lublin

1 sierpnia br. Pan Andrzej Stachnik objął stanowisko Prezesa Zarządu Biomed-Lublin. Rada Nadzorcza powierzyła mu zadanie opracowania i wdrożenia nowej strategii firmy. Andrzej Stachnik jest absolwentem Wydziału Medycyny Weterynaryjnej Uniwersytetu Przyrodniczego w Lublinie oraz uczestnikiem programu MBA Wyższej Szkoły Handlu i Finansów Międzynarodowych w Warszawie im. Fryderyka Skarbka. Posiada wieloletnie doświadczenie w branży farmaceutycznej. W latach 1991–2007 pełnił funkcję Prezesa Zarządu ACP Pharma, w latach 2007–2008 był zatrudniony na stanowisku Dyrektora ds. Rozwoju Międzynarodowego przez holenderską Grupę Kapitałową Mediq. W latach 2009–2016 Członek Zarządu i Dyrektor Handlowy firmy farmaceutycznej SymPhar Sp. z o.o. Pełni funkcję Prezesa Związku Pracodawców Hurtowni Farmaceutycznych. (*kk http://www.biomed.lublin.pl/, 24.08.2016*)

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