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Solubility of commercial octacosanol in organic solvents and their correlation by thermodynamic models at different temperatures

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ABSTRACT

Octacosanol is a high-molar-mass primary aliphatic alcohol that has shown promising effects in diabetes treatment and in lowering cholesterol. Although this compound has been obtained from non-saponifiable matter of vegetable materials by extraction with solvents, there are no solubility data in the literature. This study presents experimental values for commercial octacosanol solubility in three pure solvents (1-pentanol, 1-hexanol, and toluene) from 298.2 to 333.2 K. Solubility obtained in this study ranges from 0.0006 to 0.0602 in mole fractions. In addition, solid–liquid equilibrium results were correlated using four thermodynamic models: Van Laar, three-suffix Margules, NRTL, and UNIQUAC. Results show that high temperatures and the use of alcoholic solvents with the longest carbon chain or hydrocarbons are required to maximize commercial octacosanol solubility. The UNIQUAC model provides the best performance in the correlation of the experimental values; however, all thermodynamic models could be used to describe commercial octacosanol solubility.

1. Introduction

Policosanol is a mixture of long-chain primary aliphatic alcohols containing 20–36 carbons that is commonly found in non-saponifiable matter such as sugar cane wax and beeswax [1]. Another source of this compound is the non-saponifiable matter of vegetable oils, some of which, such as perilla seed, grape seed and rice bran oils, can provide high quantities of this compound; oils of corn germ, sesame and soybean present lower amounts of policosanol [2].

The main component of policosanol (octacosanol) can be used as a feed supplement for athletes since it improves stamina and, consequently, exercise capability [3,4]. On the other hand, the effectiveness of policosanol as a lipid-lowering agent in hypercholesterolemia treatment is still under debate [5–7]. In this context, it is important to highlight that the mixture of higher aliphatic alcohols in policosanol can show variation, so the results of clinical trials can diverge [8].

Since long-chain primary aliphatic alcohols can be obtained using solvent extraction, it is important to have knowledge of its solubility in different solvents. However, octacosanol solubility data are not found in the literature. Presented in this work are experimental values for commercial octacosanol solubility in three pure solvents (1-pentanol, 1-hexanol and toluene) from 298.2 K to 333.2 K. The solid–liquid equilibrium is correlated using four thermodynamic models, Van Laar, Margules three-suffix, NRTL and UNIQUAC, allowing the evaluation of model performance.

2. Experimental

2.1. Reagents

Commercial octacosanol was supplied by Huzhou Nanxun Shengtao Botanical Co. Ltd. (Shuanglin, Huzhou, China). 1-Pentanol (71-41-0, Merck, Germany), 1-hexanol (111-27-3, Merck, Germany), toluene (108-88-3, M-Tedia, USA), and chloroform (67-66-3, Sigma-Aldrich, USA) were used as solvents, without further purification.

The following long-chain primary aliphatic alcohols were used as standards in the characterization of commercial octacosanol:

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1-docosanol (661-19-8), 1-tetracosanol (506-51-4), 1-hexacosanol (506-52-5), 1-octacosanol (557-61-9), and 1-triacontanol (593-50-0), all of them purchased from Sigma-Aldrich (USA). The source and purity of all the chemicals are shown in Table 1.

2.2. Gas chromatography–mass spectrometry (GC–MS) analyses

Commercial octacosanol was characterized in terms of long-chain aliphatic alcohols following the methodology suggested by Asikin et al. [9], which is able to identify and quantify long-chain primary aliphatic alcohols.

GC–MS analyses were carried out on a Shimadzu QP2010Plus (Shimadzu Corporation, Kyoto, Japan) system equipped with an AOC-20i auto sampler. The column consisted of Rtx-5MS (Restek Co., Bellefonte, PA, USA) fused-silica capillary (length = 30 m, i.d. = 0.25 mm, and film thickness = 0.25 μm). The electron ionization (EI-MS) mode at 70 eV was employed. Helium (99.999%) at a constant flow of 1.02 $mL \cdot min^{-1}$ was the carrier gas. The injection volume was 1 μL (split ratio of 1:10). The injector and the ion source temperatures were set at 563 K and 473 K, respectively. The oven temperature was initially set to 423 K, increased to 593 K at 4 $K \cdot min^{-1}$, and then maintained at 593 K for 15 min. The mass spectra were registered with a scan interval of 2.0 s in the mass range of (45–500) Da.

2.3. Identification of the chemical constituents of commercial octacosanol

Commercial octacosanol components were identified on the basis of their retention time relative to a homologous of five

Table 1
Source and purity of chemicals.

Chemical Name	Source	Mass fraction purity	Purification method	Analysis method
Octacosanol	Shengtao Botanical Co. Ltd	0.9483	None	GC–MS[a], NMR[b], GC-FID[c]
1-Pentanol	Merck	≥0.98	None	GC-FID[c]
1-Hexanol	Merck	≥0.98	None	GC-FID[c]
Toluene	M-Tedia	≥0.99	None	GC-FID[c]
Chloroform	Sigma-Aldrich	≥0.99	None	GC-FID[c]
1-Docosanol	Sigma-Aldrich	≥0.98	None	GC-FID[c]
1-Tetracosanol	Sigma-Aldrich	≥0.99	None	GC-FID[c]
1-Hexacosanol	Sigma-Aldrich	≥0.97	None	GC-FID[c]
1-Octacosanol	Sigma-Aldrich	≥0.99	None	GC–MS[a], NMR[b], GC-FID[c]
1-Triacontanol	Sigma-Aldrich	≥0.98	None	GC-FID[c]

[a] Gas Chromatography coupled with mass spectrometry.
[b] Nuclear magnetic resonance.
[c] Gas Chromatography with flame ionization.

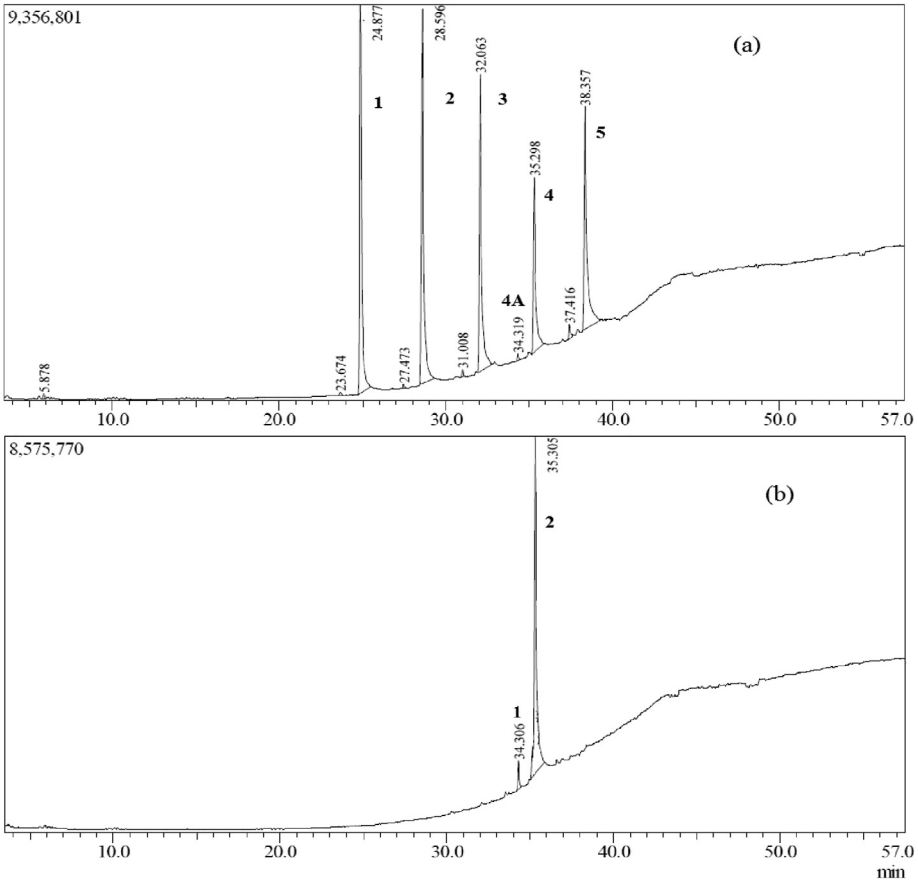

Fig. 1. Chromatograms for (a) the mix of five long-chain primary aliphatic alcohols standards identified as: **(1)** 1-docosanol (C22); **(2)** 1-tetracosanol (C24); **(3)** 1-hexacosanol (C26), **(4A)** octacosanal, **(4)** 1-octacosanol (C28), and **(5)** 1-triacontanol (C30); (b) compound used in this study with peaks identified as: **(1)** octacosanal; **(2)** 1-octacosanol.