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Modeling of the Recovery of Citric Acid Using Aliquat 336 in Natural Diluents

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Abstract: ***Reactive extraction of citric acid was studied using Aliquat 336 in various natural diluents. Distribution data was presented and compared with two modeling approaches: mass action law modeling and, differential evolution approach. Equilibrium complexation constants and stoichiometric association were obtained by the mass action law modeling. The extraction efficiency of the diluents follows the trend: soya-bean oil > sun-flower oil > sesame oil > rice bran oil. Different evolution approach was solved by program build up in Matlab and stoichiometry of association and complexation were evaluated for each data point.*** $K_D^{experimental}$ ***and*** K_D^{model} ***were compared and model was found to fit the data significantly well.***

Keywords: Aliquat 336, Citric acid, Differential evolution, Diluent, Mass action law, Reactive extraction

1. Introduction

Citric acid, natural component of many citrus fruits, can be efficiently produced by bio-route. The acid is widely used in food application, cleaning applications and in personal care. Fermentation is cheaper and globally accepted production method for citric acid production. The substrate is sucrose or glucose containing medium such as molasses, corn sleep liquor or any other inexpensive sugary solution.

The important parameters for separation of citric acid from fermentation broth have been pH and temperature. Low pH is effective for production and temperature is usually at room temperature. Thus, recovery method is required which is efficient at low pH and at room temperature. Classical method (precipitation) is involving addition of milk of lime to transform the acid to tri-calcium citrate tetra hydrate, which is removed by addition of sulfuric acid. This is a complicated and long recovery process which produces wastes in form of organic residues and calcium sulfate. Thus, there is a search of alternate recovery technique.

Compared to the various method employed for the acid recovery, reactive extraction is a closed-loop process which with proper combination of extractant (reactive component) and diluent (non-reactive component) could be effective technology in terms of high selectivity, effectiveness at low pH, low cost, easy regeneration, and high yield. Further, it is needed the solvent combination should be effective both at low and high pH since low is effective for citric acid production and high is effective for microorganisms' growth. In this regard, Aliquat 336 has been a viable candidate. The extractant reacts with the solute in the aqueous phase and is solubilized in the organic phase. So the effectiveness of the type of diluent to accommodate the complexes is also desirable.

A number of patents are available in this context [1, 2]. With one major exception, none of these appear to have been practiced commercially. The exception is a citric acid extraction process using a tertiary amines extractant and back extraction into water at higher temperature [3].

Wennerston, [4] found that tertiary amines are effective extractants for citric acid. Alamine 336 dissolved in a non-polar diluent was a suitable solvent, and the pilot plants runs showed that the process is technically feasible. Citric acid in fermentation medium was extracted with a solvent containing 30-40% butyl-acetate and 60-70% of an N,N-disubstituted alkylamide (tri-vial name '2351'). Partition equilibrium was achieved within five minutes. Approximately 96.6% citric acid was present in the organic phase after 5 stages of counter current extraction at 10-20 ˚C. Citric acid was back extracted into water at 60-80˚C. Emulsification was inhibited by the addition of activated carbon and NaCl.

Sirman et al., [5] extracted citric acid from aqueous solution using a supported liquid membrane. Bauer et al. [6, 7] used a combination of extraction/re-extraction process using a tertiary amine containing solvent was developed in this regard. Various mathematical models of amine extraction of tri-basic weak acid were developed and compared with equilibrium data on the distribution of citric acid between water and solution of trialkylamine in methyl-iso-butylketone (MIBK). It has been found that two suggested mechanisms of extraction (distribution of non dissociated portion of acid x distribution of dissociated portion of acid) are equivalent as far as the fit of the model is concerned. They only differ in the values of equilibrium constants obtained.

Few studies were done to observe the synergistic extraction of carboxylic acids. The presence of more than one acid in the aqueous phase affects the extraction characteristics. This was demonstrated by Juang et al. [8], who used solvent extraction and supported liquid membranes for the separation of lactic and citric acids with tri-n-octylamine (TOA) used as the extractant.

Juang et al., [9] studied the rate of extraction of citric acid from aqueous solution with tri-n-octylamine dissolved in xylene using a stirred membrane-based cell. They found that under the conditions studied, both the forward and the back extraction processes were mainly controlled by chemical reaction occurring at the interface on the organic side. The intrinsic rate constants for the formation and

dissociation of the acid complex were determined and a possible reaction mechanism was proposed.

Nikhade et al., [10] reported on the extraction and kinetics of citric acid from aqueous solutions with Alamine 336 using with methyliso-butylketone (MIBK) as diluent. The theory of extraction accompanied by a chemical reaction has been used to obtain the intrinsic kinetics of extraction of citric acid by Alamine 336 in MIBK. The reaction has been found to be first order in both Alamine 336 and citric acid with a rate constant of 0.013 m^3/kmol.s.

In the present work it has been tried to use Aliquat 336 with variety of natural diluents to find the effectiveness of the reactive extraction process for the recovery of citric acid. The results were modeled by mass action equilibria model as well as differential evolution technique. Differential evolution algorithm has been prepared. The model and experimental observations were compared and a good agreement was observed.

2. Materials and Methods

Aliquat 336 (Methyltricaprylammonium chloride, $C_{25}H_{54}NCl$) (molar mass: 404.17 g/mol; density: 0.889 g/cm^3), a quaternary amine was used as an extractant. Citric acid (molar mass 210.13; density 1.542 g/cm^3; Himedia India. Ltd.), diluents are of technical grade and were used without pretreatment. Sodium hydroxide used for titration is of analytical grade and was supplied by Ranbaxy, India. For the standardization of the sodium hydroxide, oxalic acid (99.8%) was obtained from S. d. fine–Chem. Ltd, India. Phenolphthalein solution (pH range 8.2–10.0) was used as indicator for titration and was obtained from Ranbaxy, India.

3. Theory

Chemical extraction systems make use of liquid ion exchangers, more commonly called as extractants or reactants, to perform selective separations. The distribution data can be interpreted by a set of equilibria involving the formation of complexes with “n” citric acid molecules and “m” amine molecules.

Distribution of non-dissociated acid

$$nH_3A + m(B)_{org} = ((B)_m H_{3-m} A)_{org} \quad m= 1, 2, 3 \quad (1)$$

Both sets of equations are characterized by the thermodynamic extraction constant

$$K_E = \frac{[(B)_m (H_3A)_n]_{org}}{[H_3A]^n_{aq}[B]^m_{org}} \quad (2)$$

If assuming n=1, the above the distribution coefficient can be defined in terms of equilibrium complexation constant as

$$K_D = \frac{[(B)_m H_3A]_{org}}{[H_3A]_{aq}} = K_E[B]^m_{org} \quad (3)$$

Taking log

$$\log K_D = \log K_E + m[B]_{org} \quad (4)$$

The loading of the extractant z, is defined as the total concentration of acid in the organic phase divided by the total concentration of amine in the organic phases.

$$z = \frac{[H_3A]_{org}}{[B]_{org}} \quad (5)$$

If the organic phase is not highly concentrated, i.e. at very low loading, the (1:1) complex is formed and this happens when z<0.5. The equilibrium complexation constant value for (1:1) complexation can be found using the following equation [3]

$$z/1-z = K_{E(1:1)}[H_3A]_{aq} \quad (6)$$

If z values are higher than 0.5, other types of complexations are considered such as (1:2), (1:3) etc. Usually in citric acid extraction other types of aggregates such as 2:3, 3:2 etc also needs to be considered because such types of complexations are also contributing to the overall loading ratio.

4. Results and Discussion

4.1 Optimum concentration of Aliquat 336 for chemical extraction of citric acid.

In the present text, Aliquat 336 has been used with rice bran oil, sunflower oil, soya-bean oil and sesame oil, respectively. Extractant was used in the volume percentage of 10 to 30 % in diluents. Higher concentration were also checked with these natural diluents (Figure 1) but employing Aliquat 336 greater than 30% could not result in significant increase in distribution of acid. Further this also would result in increase of the cost of the operating unit. Higher concentration of extractant is also not advisable owing to two reasons. First higher the concentration of these extractants, higher is its effect in terms of toxicity to the microorganisms if recovery is to be done from bio reactor. Secondly, most of the extractants are active only in acidic conditions and with the raise of pH the recovery falls very largely [11] and the presence of large excess of extractant would not participate in extraction process.

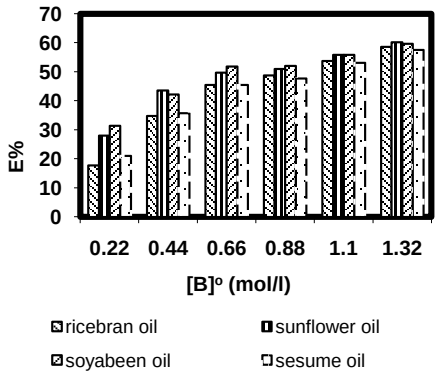

Figure 1: Finding the optimum Aliquat 336 concentration

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4.2 Extraction of citric acid using Aliquat 336 in natural diluents

Acid concentration was varied from 0.1 to 0.8 mol/l for Aliquat 336 concentration of 0.22, 0.44 and 0.66 mol/l in different diluents. Figure (2) shows the effect of acid concentration on the extractability by Aliquat 336 in sunflower oil. Similar plot have been obtained for other natural diluents. For extraction of citric acid using Aliquat 336 in different diluents, K_D was found to decrease with increase in acid concentration; however the decrease was marginal for lower concentrations of acid 0.1-0.4 mol/l whereas for higher acid concentration (0.6 and 0.8 mol/l) the decrease was great. Thus reactive extraction is more successful in separation of acid from dilute solutions. Initially as the acid shall be produced in the bioreactor, its concentration shall be low and hence the success of using reactive extraction technology for acid separation is justified.

Among the various diluents used, better solvation of Aliquat 336-acid complexes were found in the case of soya-bean oil with E% as high as 51.81%. The trend of extractability of the acid using Aliquat 336 in various diluent is as: soya-bean oil ($E\%_{Bo=\ 0.22}$ = 31.41-11.39%; $E\%_{Bo=\ 0.44}$ = 42.18-18.33%; $E\%_{Bo=\ 0.66}$=51.81-25.83%) >sunflower oil($E\%_{Bo=\ 0.22}$ = 27.97-12.58%; $E\%_{Bo=\ 0.44}$ =43.61-18.85%; $E\%_{Bo=\ 0..66}$=49.75-25.65)>sesame oil ($E\%_{Bo=\ 0.22}$ = 21.02-5.77; $E\%_{Bo=\ 0.44}$ = 35.73-11.22%; $E\%_{Bo=\ 0.66}$=45.53-14.49)>rice bran oil ($E\%_{Bo=\ 0.22}$ = 17.73-10.12%; $E\%_{Bo=\ 0.44}$ = 34.83-18.76%; $E\%_{Bo=\ 0.66}$=45.51-23.74). Thus it could be suggested that Soya–bean and sunflower oil are better solvents for reactive extraction of citric using Aliquat 336.

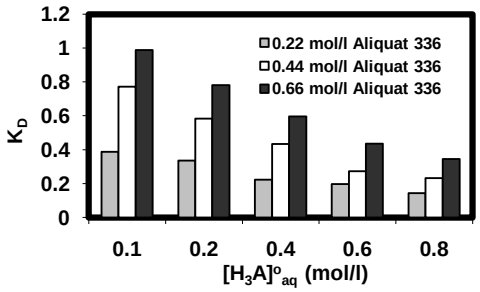
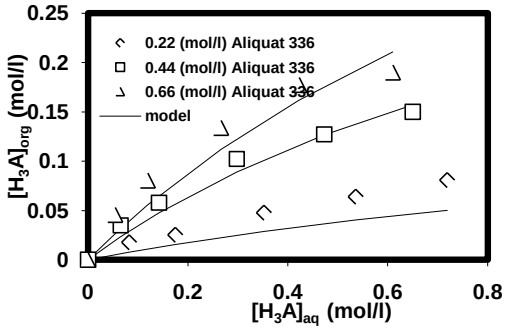


Figure 2: Effect of acid and Aliquat 336 concentration on the reactive extraction of citric acid using Aliquat 336 in sunflower oil

Figure (3) shows the equilibrium curves showing the effect of Aliquat 336 concentration on the extractability of acid. Aliquat 336 was employed in four different diluents; rice bran oil, sunflower oil, soya–bean oil, sesame oil, respectively. It could be easily seen that in all the diluents employed, higher is the percentage of Aliquat 336, higher in the extraction obtained at all the acid concentration studied (0.1–0.8 mol/l). This suggests that it shall be better to use higher percentage of Aliquat 336. The average increase in K_D values when Aliquat 336 concentration was increased from 0.22 to 0.44 mol/l was 63%, 47%, 47%, 61% for when rice bran oil, sunflower oil, soya-bean oil, sesame oil was used as diluents, respectively. However, since extraction of citric acid is mostly to be done from bioreactor, where the problem of toxicity is of major concern, it is preferable to use extractant is low concentrations. Further, Aliquat 336 is highly viscous (μ = 1500 Pa.s at 30 °C) so in any case its volume percentage above 30 % could create problems of three phase formation in inert diluents. So in the present study only volume percentages of 10 to 30% were employed. In some cases where the three phase formation was encountered, the two phases were separated by centrifugation at 10,000 rpm for 10 minutes.

a)



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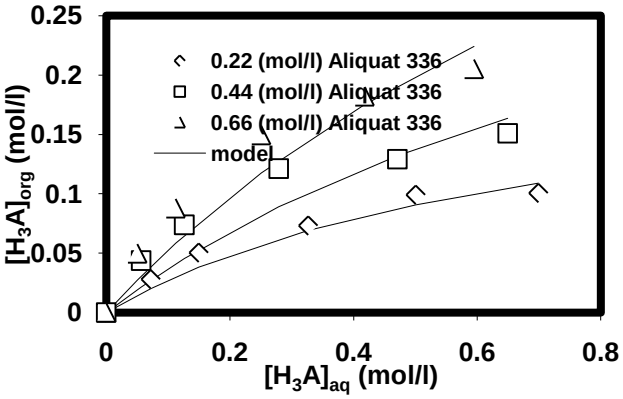

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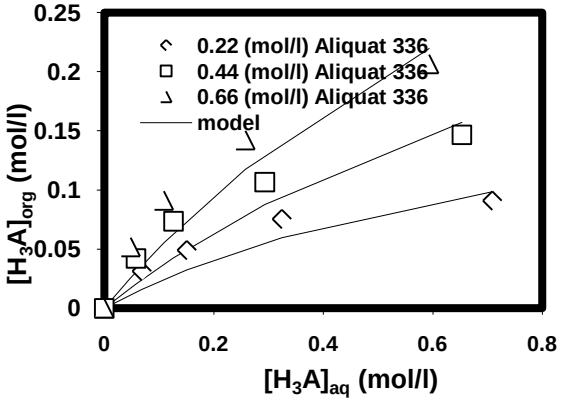

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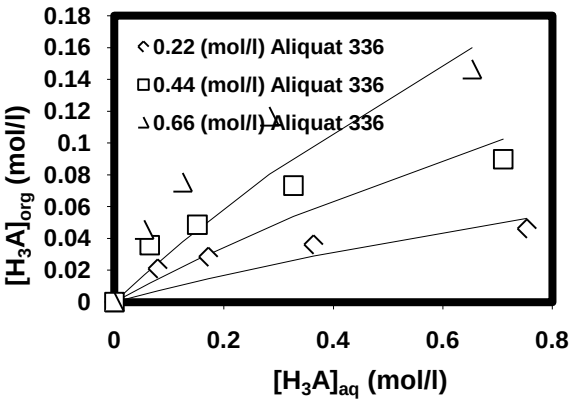

Figure 3: Equilibrium curves for reactive extraction of citric acid (0.1-0.8 mol/l) using Aliquat 336 (10-30%) a) rice bran oil, b) sunflower oil, c) soyabeen oil and d) sesame oil, respectively

Experiments involving 30% Aliquat 336 concentration and lower acid concentrations provided the highest degree of extractions. This extraction could have raised the pH of the solution. So even if excess amount of Aliquat 336 is present it would have gone without recovering anything and hence additions cost would have to be faced for recycling this Aliquat 336 back to the broth. So it is optimum to use 30 % Aliquat 336. This is also justified in view of the problem of toxicity it could raise. Further it has been mentioned that distribution coefficient of acid using various concentration of amine goes through a maxima [12, 13]. The reason for this being that Aliquat 336 in alone is a relatively poor solvation media for the complexes.

Citric acid is a tri-carboxylic acid and has the tendency to interact with 1-3 extractant molecules. In some cases however a single extractant molecule can complex with more than one acid molecule or form aggregates and hence could result in higher loading ratio values. If z>0.5, it is expected that extractant exhibits higher acid-amine complexations such as 1:2 or 1:3, whereas if it is low only 1:1 complexations dominates. Solid lines in Figure (4) show the model values of the data. It can be seen that in the extraction of citric acid using Aliquat 336 in rice bran oil and sunflower oil there has been a good fit of experimental data by the model values.

Figure (5) represents the loading curves for extraction of citric acid using Aliquat 336 in various diluents respectively. In most of the cases loading was lower than 0.5, hence suggesting that the association is 1:1.

4.3 Equilibrium complexation constant using law of mass action modeling

Equilibrium complexation constant (K_E) determine the complexation ability of a particular extractant. Since solvation is the major criterion that decides the amount of acid extracted with the particular extractant, K_E shall be the deciding parameter for choosing a particular diluent with the extractant. There are three types of K_E values:

i) Overall complexation constant for reactive extraction using Aliquat 336 in natural diluents

Plot of Eq. (4) i.e. the plot of log K_D versus amine concentration could predict the number of molecules of Aliquat 336 associated with one molecule of solute and equilibrium complexation constant value. A good fit of Eq. (4) gives a good fit of the experimental data, still the values of stoichiometric complexation constant (m) has been found to be negative, suggesting that more than one Aliquat 336 complexes with the acid molecule. K_E values were found to be 1.41, 2.36, 3.29 and 1.84, 3.78, 3.44 and 3.12, 3.97, 3.86 and 10.88, 9.45, 0.97 for Aliquat 336 in rice bran oil, sunflower oil, soya-bean oil, sesame oil for 10%, 20%, 30%, respectively.

ii) From loading ratio considering 1:1 complexation for reactive extraction using Aliquat 336 in natural diluents

Kertes et al., [3] predicted that the plot of z/(1–z) against acid concentration in the aqueous phase could predict the equilibrium complexation constant value for (1:1) complexation provided loading ratio value (z) is less than 0.5. In the studies on natural diluents, z values were less than 0.5 and hence on 1:1 complexation are dominating. K_E(1:1) values for reactive extraction of citric acid using Aliquat 336 in rice bran oil, sunflower oil, soya-bean oil, sesame oil were found to be 0.796, 1.4, 1.147, 0.416 and 0.854, 0.910, 0.852, 0.427 and 0.77, 0.86, 0.842, 0.489 l/mol for 10%, 20% and 30% extractant in diluent respectively. Solid lines in Figure 3 were drawn by using these model values.

4.4 Differential Evolution Modeling

Global optimization is necessary in field of engineering. For problems, such as non-differentiable, non-linear, multi-dimensional or have many local minima, which are difficult to solved mathematically optimization technique can be really useful. Differential Evolution (DE) is a population based optimization technique that tries to improve solutions of a given objective function with respect to given measure of quality. It is based on Genetic Evolution methodology in which Global optimum is determined.

Population of points within the range of the function to be maximized or minimized is used to search for the optimum. Assume P_n= size of population and D = Dimension of population. Target vector is selected from one out of these P_n vectors. Two other vectors from the same population are selected at random and their difference is evaluated, this difference is vectorial in nature. Weight factor 'W' which is specified initially is then multiplied to this difference and result is added to a third vector which is again randomly selected. The result is called noisy random vector. Then Crossover is done between target vector and noisy random vector and the result is called trial vector. Both trial vector and noisy random vector is evaluated for objective function and the winner is replaced by the other one in the population. This process is carried on till a sufficient convergence criterion is obtained [14–18].

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Steps performed while exercising Differential evolution is explained below with the help of Figure (19) [18]

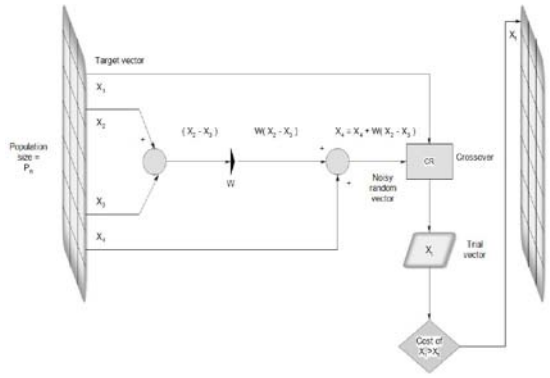

Figure 7: Steps performed while exercising Differential evolution

Table 1 shows the value of model parameters (a, b and K_E) along with the model K_D values. The experimental K_D and model K_D shows slight deviation (less than 5%) in all cases. Thus differential evolution modeling approach could successfully evaluate the model values.

5. Conclusion

Experimental and modeling approach was followed to find the equilibrium and kinetic data for reactive extraction of citric acid using Aliquat 336 in various diluents. Chemical extraction using Aliquat 336 was studied for acid concentration in the range of 0.1-0.8 mol/l in natural diluents (sunflower oil, soya-bean oil, rice bran oil and sesame oil, respectively). K_D was found to decrease with increase in acid concentration. Better solvation of Aliquat 336-acid complexes were found in the case of soya-bean oil with E% as high as 51.81%. In all the diluents employed, higher is the percentage of Aliquat 336, higher is the extraction obtained at all the acid concentrations studied. In most of the cases loading was lower than 0.5, hence suggesting that the association is 1:1. Equilibrium complexation constant was obtained using mass action law equilibria modeling. K_E values were found to be 1.41, 2.36, 3.29 and 1.84, 3.78, 3.44 and 3.12, 3.97, 3.86 and 10.88, 9.45, 0.97 for Aliquat 336 in rice bran oil, sunflower oil, soya-bean oil, sesame oil for 10%, 20%, 30%, respectively. 1:1 equilibrium complexation constant was also evaluated using theory and K_E values were reported. Algorithm for differential evolution modeling was generated and the approach was used to model the K_E and stoichiometric association constant values. Though the use of natural diluents makes the process of reactive extraction environment-friendly; however, there is a scope to explore this field at different isothermal conditions with binary extractants and/or diluents to estimate the parameters which can intensify the process.

Table 1: Differential evolution model parameters and comparison of model K_D obtained with the experimental K_D

$[H_3A]_o$	$[B]_o$	Diluent	Model Parameters				K_D^{exp}
			a	b	K_E	K_D	
0.1	0.22	rice bran oil	0.99	1.1	1.10	0.20	0.22
0.2			0.94	1.0	0.78	0.13	0.14
0.4			0.99	1.1	0.83	0.13	0.14
0.6			0.55	1.2	0.82	0.11	0.12
0.8			0.39	1.2	0.97	0.11	0.11
0.1	0.44		0.97	1.5	1.31	0.50	0.53
0.2			0.89	1.5	1.08	0.40	0.41
0.4			0.90	1.6	1.31	0.33	0.34
0.6			0.94	1.6	1.36	0.24	0.27
0.8			0.34	1.4	0.80	0.19	0.23
0.1	0.66		0.98	1.2	0.80	0.84	0.84
0.2			1.00	1.9	1.16	0.64	0.67
0.4			-	1.6	0.96	0.50	0.5
0.6			1.00	1.5	0.82	0.38	0.42
0.8			0.94	1.4	0.68	0.30	0.31
0.1	0.22	sunflower oil	0.99	1.2	2.12	0.40	0.39
0.2			1.00	1.2	2.20	0.34	0.34
0.4			0.94	1.1	1.49	0.22	0.22
0.6			0.73	1.1	1.72	0.20	0.2
0.8			0.86	1.2	1.59	0.13	0.14
0.1	0.44		1.00	1.6	1.97	0.76	0.77
0.2			0.99	1.5	1.65	0.52	0.58
0.4			0.95	1.6	1.80	0.40	0.43
0.6			0.59	1.5	1.03	0.26	0.27
0.8			0.73	1.2	0.80	0.23	0.23
0.1	0.66		1.00	2.4	1.64	0.97	0.99
0.2			0.98	2.3	1.53	0.76	0.78
0.4			0.62	2.4	1.23	0.52	0.6
0.6			1.00	1.8	1.12	0.42	0.44
0.8			0.72	1.5	0.77	0.31	0.35
0.1	0.22	soya-bean oil	1.00	1.2	2.46	0.44	0.46
0.4			0.87	1.2	1.78	0.23	0.23
0.8			0.74	1.2	1.06	0.10	0.13
0.1	0.44		0.99	1.6	1.89	0.69	0.73
0.2			0.93	1.6	1.64	0.56	0.58
0.4			0.92	1.3	1.08	0.33	0.36
0.8			0.76	1.2	0.75	0.22	0.22
0.1	0.66		1.00	2.5	1.77	1.06	1.08
0.2			1.00	2.1	1.50	0.80	0.84
0.4			0.91	1.9	1.10	0.48	0.55
0.8			-	1.5	0.80	0.33	0.35
0.1	0.22	sesame oil	0.97	1.2	1.40	0.25	0.27
0.2			1.00	1.2	1.83	0.15	0.17
0.4			0.93	1.1	0.60	0.09	0.1
0.8			0.72	1.1	0.35	0.05	0.06
0.1	0.44		0.94	1.6	1.39	0.52	0.56
0.2			0.95	1.6	1.06	0.31	0.32
0.4			0.88	1.6	0.77	0.20	0.22
0.8			0.75	1.2	0.37	0.12	0.13
0.1	0.66		0.98	2.4	1.43	0.83	0.84
0.2			0.91	2.2	1.07	0.57	0.6
0.4			1.00	1.6	0.77	0.39	0.42
0.8			0.94	1.4	0.34	0.16	0.17

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