Kinetics and thermodynamics of cottonseed oil extraction

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1. INTRODUCTION

Cotton has been grown globally for centuries mainly to meet the basic requirements of cotton fabrics, whereas cottonseed left after the removal of the cotton fiber is also a valuable by-product. Cottonseed is a rich source of oil and protein and India is the second largest producer of cotton seed which is the sixth largest source of oil available in the world (Shekhar, 2004). Cottonseed oil is important for food processing due its distinct properties. It is rich in antioxidant tocopherol (Vitamin-E) which resists rancidity development and contributes to its stability, giving the product a longer shelf life. It is described as a “naturally hydrogenated oil” as it contains sufficient natural palmitic, stearic and mysterious acids. Due to the presence of unsaturated oleic, linoleic and linoleinic fatty acids it is also known as a “Heart Healthy Oil”. It is used as salad oil, for the frying of potato and corn chips, in seafood and oriental foods. However, it is now being used in a much wider range of processed foods including cereals, bread and snack foods (Brien et al., 2005). It also has non food applications such as in bio-diesel production (Junfeng et al., 2010), and as a lubricant additive (Ertugrul et al., 2004).

The oil can be obtained from cottonseeds by mechanical pressing, solvent extraction, and supercritical fluid extraction (Liauw et al., 2008; Bhattacharjee et al., 2007; List et al., 1984). The quality of oil extracted by mechanical pressing or squeezing in an expeller press is inferior and has a limited recovery of only 94-95%. Though the supercritical extraction of flaked cottonseed with carbon dioxide at 323-353K and 8,000-15,000psi pressure yields a better quality oil with less refining loss, operating and investment costs are very high compared to those for conventional solvent extraction or mechanical pressing.

The solvent n-hexane has been extensively used for oil extraction from oilseeds because of its low boiling point of 336.16–342.26K, high stability, low corrosiveness, low greasy residual effect, better odor and improved flavor for milled products containing cottonseeds and oil. However, it is highly toxic and hazardous. Petroleum derived n-hexane is commercially used in solvent extraction plants due to its higher extraction efficiency in spite of the fact that it is graded as highly toxic and hazardous. The present study is based on the use of both a much safer and non toxic solvent ethanol and n-hexane for the extraction of oil from cottonseed. The extraction data were obtained by varying temperature, solvent-solid ratio and particle size, to compare the extraction efficiency of both ethanol and n-hexane. The data show that nearly the same amount of extraction is possible at a higher solvent-solid ratio for both the solvents. This study has established that the kinetics of oil extraction follows a second order reaction mechanism. The thermodynamic analysis of the data shows that both $\Delta H^\circ$ and $\Delta S^\circ$ are positive, and $\Delta G^\circ$ is negative indicating that the extraction process is endothermic, irreversible, and spontaneous.

(Johnson and Lasus, 1983; Becker, 1978). It is listed as a hazardous air pollutant by the US Clean Air Act since it can react with other pollutants to produce ozone and photochemical oxidants (Wan et al., 1995a; Hammoungjai et al., 2000) and its use in oilseed extraction plants can adversely affect workers’ central nervous system (Becker, 1978; Johnson and Lasus, 1983; Lasus et al., 1991; Gandhi et al., 2003). The control of its emission levels in extraction plants requires the installation of additional control equipment at additional costs (Conkerton, et al., 1995). Seth (2007) has reported that the use of n-hexane in small capacity plants makes the process expensive because of high operating losses (typically ranging from 0.75 to 7.5 L/MT of processed seeds). Further, n-hexane, being a petroleum product, faces occasional scarcity and fluctuation in price depending on the supply and demand of gasoline.

The cottonseed oil extraction industry is seeking a safer alternative solvent to eliminate the emissions of n-hexane into the atmosphere as well as potential traces of n-hexane in both the refined edible oils and the meal. The prevention of food adulteration act in India has ruled that the oil obtained by solvent extraction shall not contain more than 5ppm of n-hexane and the meal should not contain more than 10ppm (PFA Act, 1954). It is, however, desirable that hexane be removed completely from the meal. Most of the n-hexane extraction units suffer from the difficulty of totally removing residual solvents from the meal (Eskin et al., 1995).

The extraction of oil with aqueous solutions (acidic or alkaline at 318 to 258K) has been reported in the literature as an alternative to organic solvents (Hagenmaier et al., 1973; Kim, 1989; Hammoungjai et al., 2000). However, due to the low selectivity of aqueous solutions for lipids, the simultaneous extraction of proteins, carbohydrates and other compounds also occurs in this case. Aqueous extraction is also limited by (i) the lower efficiency of oil extraction, (ii) the need for an additional de-emulsification step to recover the oil and (iii) the production of an aqueous effluent which requires further treatment (Hammoungjai et al., 2000). Heptane and iso-hexane (Wan et al., 1995a; Wan et al., 1995b; Conkerton, et al., 1995, Kuk and Hron, 1998), methylpentanes (Ayer and Scott, 1951), acetone (Frampton et al., 1967; Kuk et al., 2005) and alcohols (Abraham et al., 1988; Hron et al., 1994; Sineiro et al., 1998) are also reported as other solvents besides aqueous for oil extraction.

Isopropanol and ethanol are the most promising solvents for oil extraction from cottonseed (Lasus et al., 1991; Abraham et al., 1988; Hron et al., 1994, Bhowmick, et al., 2003), sunflower seed (Sineiro et al., 1998), soybean (Baker and Sullivan, 1983), Quercus suber L. fruits (Ferreira-Dias et al., 2003), and Neem (Liauw et al., 2008). Ethanol has, however, emerged as a better alternative solvent since its cost is low and it can be produced from a large variety of biological materials through fermentation. Although flammable (flash point = 282.06K; ignition temperature = 698.16K), it is rated as safe (Hron et al., 1994), non-toxic and has lower handling risks compared to n-hexane (flash point = 250.16K; ignition temperature = 498.16K). Ethanol provides a much safer work environment in extraction plants since its threshold limit value of the time weight average is 1000 ppm as compared to 50 ppm for n-hexane [NIOSH, 2007].

The use of ethanol as an extraction solvent has the advantage of eliminating the toxicity problems in the meal for animal feedstuffs caused by n-hexane. Abraham, et al. (1988) have reported that 95% oil could be extracted with ethanol from cottonseed, and the resulting alcoholic miscella could be easily refined with caustic to produce an acceptable oil (Hron and Koltun, 1984). The use of alcohols to extract oil from seeds is not new. Japanese workers, among others, reported the use of aqueous ethanol to extract oil from soybeans as early as 1932 (Hron et al., 1994). However, the process could not be commercialized for several reasons. Cottonseed, like other oilseeds, is pre-treated before extraction to release the oil. The pretreatment operations include de-hulling, cracking, flaking, thermal treatment etc. (Hoffman et al., 1989).

The extraction rate of oil is a major influential factor for designing a large-scale solvent extraction system. Normally, extraction rate depends on the nature of the solvent and the oil, reaction time between solvent and seeds, temperature of the process, particle size of the meal, and solvent-solid ratio. Coats and Wingard (1950) observed that the oil extraction rate was largely dependent on particle size. Seth et al. (2007) reported that oil from cracked particles (grits) of soybeans, cottonseed, flaxseed, and peanuts could be extracted more easily compared to flakes of equivalent thickness.

Keeping the literature findings in mind, ethanol and n-hexane were used in the present study as solvents for the extraction of oil from cottonseeds to generate data for the study of the kinetics and thermodynamics of the extraction process. The data were generated by varying parameters such as solvent-solid ratio (SR), temperature, and particle size (PS). The results obtained for both the solvents are compared to establish the advantages of ethanol as an alternative to n-hexane.

1. MATERIALS AND METHODS

1.1. Materials

Seed Preparation

For the purpose of this study, acid delinted cottonseeds of Gossypium Hirsutum (P8-6 variety) were obtained from the Indian Agricultural Research Institute, Pusa, N. Delhi. The seeds were de-hulled in the laboratory using a mixer grinder. The de-hulled seeds were dried in a vacuum oven at 318K.
to remove moisture and then segregated into three fractions A, B, and C using standard sieves. The average particle size of the three fractions A, B, and C were 0.6, 1.6 and 2.4 mm respectively. The de-hulled seeds were found to contain 36.87% oil on a dry solid basis. The oil percentage was determined by an exhaustive extraction method using hexane in a soxhlet apparatus.

Solvants

Both n-Hexane and Ethanol of L.R grade from Fisher were redistilled and used as solvents.

1.2. Methods

Oil Testing

A) Solid Sample: The oil content in the solid sample was determined according to the method described in Appendix F of IS: 7847-1968 of the Bureau of Indian Standards [IS: 7847-1968].

B) Liquid Sample: To estimate the oil content in a liquid sample the oil percentage was determined based on the change in density of the sample. A Mettler Toledo density meter, Model DE-45 Delta Range was used to record the change in density of up to 4 decimal places. First, calibration curves were prepared using raw cottonseed oil extracted by n-hexane and ethanol respectively. A known quantity of oil was dissolved in the same solvent at different dilutions, the density of each dilution was measured and the calibration curve density vs oil concentration (gm oil/L solvent) was prepared. To determine the oil content in the liquid phase, the density of the clear liquid portion separated after centrifugation was obtained. The oil content in the sample was calculated using the calibration curve. For few representative clear liquid samples, the results were cross-checked for oil content by evaporating the solvent in a vacuum oven to obtain solvent-free oil (Conkerton et al., 1995).

Oil extraction

The weighed quantity of cottonseed samples taken in the airtight 30 ml plastic bottles and the solvent bottle were kept overnight in a constant temperature incubator to attain the desired uniform temperature. The measured quantity of solvent was then mixed with the seed sample in the bottles for oil extraction. After mixing, the bottles were closed airtight and placed immediately in the shaking incubator. The study was conducted for a period of three hours and one plastic bottle was taken out after a specified period of 1, 5, 10, 20, 40, 60, 120 and 180 minutes; the supernatant liquid (slightly hazy) was removed from the plastic bottle and centrifuged at 9000rpm for 5 minutes. The clear transparent liquid obtained after centrifuge was removed for oil determination. Extraction was conducted at four temperatures i.e. 288, 298, 308, and 318±1K, three solvent-solid ratios (5:1, 10:1 and 15:1) and three particle sizes i.e., A, B and C. Percentages of oil extracted was calculated using Eq.1. Experiments were duplicated and the average value was taken to ensure accuracy and the results were expressed on a dry basis.

\[
\text{Percent Oil Extracted (\%)} = \frac{C_t \times V}{w_0} \times 100
\]

Where: \( C_t \) = Oil concentration in solvent at any time, \( t \) (g/L), \( V \) = Volume of solvent mixed with seed sample (L), \( w_0 \) = Weight of total oil present in the seed sample (g)

2. RESULTS AND DISCUSSION

2.1. Effect of Particle Size

The oil extraction was carried out with the seed sizes A (0.6 mm), B (1.6 mm) and C (2.4 mm) using both the solvents at 308K and solvent-solid ratio 10:1 for three hours to study the effect of particle size. The results are shown in Figure 1. It may be seen from Figure 1 that the oil extracted in 3 hrs is 85.24, 49.36 and 40.82% from the seeds of sizes A, B and C with n-hexane and 81.75, 48.07 and 40.22% from the seeds of sizes A, B and C with ethanol respectively under similar conditions. The lower rate of extraction from larger size particles B and C can be attributed to both the lower surface area per unit weight and higher resistance to extraction due to the longer path the solvent has to travel to leach out the oil as compared to A size particles.

2.2. Effect of Solvent-Solid Ratio (SR)

In the present work to study the effect of solvent-solid ratio (SR) on oil, extraction experiments were conducted at 308K with Size-A by varying SR. The SR of 5:1, 10:1 and 15:1 were used during the study. It can be seen from Figure 2 that by increasing the SR the amount of oil extracted increased. The extraction of oil at the SR of 5:1 was very low, whereas at the other two ratios of 10:1 and 15:1 the oil extraction was better. In the case
of extraction with ethanol at the SR of 5:1 the oil extraction was only 57.71%, whereas at the SR of 10:1, it was 82.31% and at the SR of 15:1, it was 86.42%. The lower oil extraction at the SR of 5:1 can be explained by the fact that the solubility of oil in ethanol may restrict the oil extraction from the seed near the saturation point of oil in ethanol. At higher SR the efficiency of oil extraction with ethanol was found to be comparable with n-hexane. At the SR of 10:1 and 15:1 the extraction was 85.24% and 88.71% with n-hexane and 82.31% and 86.42% with ethanol. Slightly lower oil extraction with ethanol can be attributed to the solubility of cottonseed oil in ethanol as compared to solubility in n-hexane.

2.3. Effect of Extraction Temperature

The studies were conducted at SR of 10:1 and particle size-A by varying the temperature from 288 to 318K to see its effect on the oil extraction process. It was found that an increase in temperature has a direct effect on the amount of oil extracted as shown in Figure-3. It may be seen from Figure-3 that the amount of oil extracted with n-hexane increased from 58.20% to 92.84% as the extraction temperature increased from 288 to 318K, whereas the increase in extraction was from 48.08% to 93.84% as the temperature increased from 288 to 318K, when ethanol was used as the solvent. This increase may be due to the increase in oil solubility in the solvent at a higher temperature and a decrease in viscosity of the solution resulting in lower diffusion resistance in the pores of the particle.

2.4. Effect of Extraction Time

The extraction of oil from cottonseeds was carried out with n-hexane and ethanol for a period of 3 hours at 308K and at SR of 10:1 and the data were plotted in Figure-4. It can be seen that the extraction of oil increases with time and the rate of extraction is initially high and then tapers off. The oil extraction in the first hour reached 77.28% with n-hexane and 75.16% with ethanol at 308K. After this there is a small increase in oil extraction and the total oil extracted in 3 hours is 85.25% and 81.75% with n-hexane and ethanol respectively at 308K. The initially high rate of oil extraction may be because of the dissolution and the scrubbing of the oil available on the surface and the later slow extraction rate may be attributed to the diffusion of the remaining oil into the solution as supported by the study of Rakotondramasy, et al. (2007).

2.5. Kinetics

Various studies have been reported to describe the mechanism and kinetics of the extraction processes. A second order reaction kinetics for the solid-liquid extraction process has been proposed by most of the researchers (Wiese et al., 1987; Meziane, et al., 2006; Meziane, et al., 2008; Rakotondramasy, et al. 2007, Sepidar et al. 2009, Wenjuan et al., 2010). It is typical of a second order process to take place in two stages. First, the major part of the solute gets extracted quickly because of the dissolution and scrubbing of the surface oil caused by the higher driving force of the fresh solvent and in the next stage where the extraction rate is slowed down by the low diffusion of the remaining oil.

The rate of dissolution of the oil into the solution can be described by the following second order rate equation:

$$\frac{dC_t}{dt} = k(C_e - C_t)^2$$

(2)
experimental data are recorded using an
spread
the experimental data by plotting
order extraction constant,
\( k \), can be calculated from

\[
C_t = \frac{C_o^2 k t}{1 + C_o k t}
\]  

(3)

The extraction rate Eq.(3) can be rearranged as follows:

\[
C_t \frac{t}{1 + C_o k t} = k C_o^2
\]  

(4)

Now, when \( t=0 \), the LHS of Eq.(4) will be initial extraction rate, \( E_i \), and the denominator of RHS will reach 1.0 thus Eq.(4) reduces to Eq.(5)

\[
E_i = k C_o^2
\]  

(5)

Thus Eq.(4) after combining with Eq.(5), the rearrangement would yield the following linear form:

\[
\frac{t}{C_t} = \frac{1}{E_i} = \frac{t}{C_o}
\]  

(6)

The initial extraction rate, \( E_i \), the concentration of oil in the solution at equilibrium, \( C_o \) and the second order extraction constant, \( k \), can be calculated from the experimental data by plotting \( t/C_t \) vs. \( t \). The experimental data are recorded using an MS-excel spread sheet and the estimated results at SR 10:1 to 15:1 and temperatures from 288 to 318K are given in Table 1. The results show that for all cases, the value of the regression coefficient (\( R^2 \)) lies between 0.98-0.99. This indicates that the data are well described by second order kinetics. Further, it can be seen that the initial rate of extraction is not appreciably affected by SR but is largely affected by temperature.

### 2.6. Thermodynamics

Thermodynamic considerations of an extraction process are necessary to conclude whether the process is spontaneous or not. The Gibbs free energy change (\( \Delta G^o \)) is an indication of the spontaneity of a chemical reaction and therefore is an important criterion for spontaneity. Reactions occur spontaneously at a given temperature if \( \Delta G^o \) is a negative quantity. Gibbs free energy (\( \Delta G^o \)), enthalpy change (\( \Delta H^o \)) and entropy change (\( \Delta S^o \)) are calculated using the following equations:

\[
K_e = \frac{C_o}{C_s}
\]  

(7)

\[
\Delta G^o = -RT \ln K_e
\]  

(8)

\[
\Delta G^o = \Delta H^o - T \Delta S^o
\]  

(9)

\[
\ln K_e = -\frac{\Delta H^o}{RT} + \frac{\Delta S^o}{R}
\]  

(10)

Where \( C_s \) is the concentration of oil in solid phase at equilibrium and could be obtained by material balance. \( R \) is the ideal gas constant (8.314 J mol\(^{-1}\) K\(^{-1}\)), and \( T \) is the temperature (K). Eq. 10

---

**Table 1**

<table>
<thead>
<tr>
<th>Solvent</th>
<th>SR (ml/g)</th>
<th>Temp., (K)</th>
<th>( C_o ) (g/L)</th>
<th>( k ) (L/g-min)</th>
<th>( E_i ) (g/L-min)</th>
<th>( R^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-Hexane</td>
<td>10:1</td>
<td>288</td>
<td>22.675</td>
<td>4.13 \times 10^{-3}</td>
<td>2.124</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>10:1</td>
<td>298</td>
<td>26.455</td>
<td>4.29 \times 10^{-3}</td>
<td>3.003</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>10:1</td>
<td>308</td>
<td>32.362</td>
<td>4.56 \times 10^{-3}</td>
<td>4.775</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>10:1</td>
<td>318</td>
<td>34.843</td>
<td>4.66 \times 10^{-3}</td>
<td>5.656</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>15:1</td>
<td>288</td>
<td>17.668</td>
<td>4.60 \times 10^{-3}</td>
<td>1.435</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>15:1</td>
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<td>20.703</td>
<td>6.44 \times 10^{-3}</td>
<td>2.762</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>15:1</td>
<td>308</td>
<td>22.573</td>
<td>8.04 \times 10^{-3}</td>
<td>4.098</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>15:1</td>
<td>318</td>
<td>23.041</td>
<td>11.15 \times 10^{-3}</td>
<td>5.917</td>
<td>0.99</td>
</tr>
<tr>
<td>Ethanol</td>
<td>10:1</td>
<td>288</td>
<td>17.921</td>
<td>5.61 \times 10^{-3}</td>
<td>1.802</td>
<td>0.99</td>
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<td></td>
<td>10:1</td>
<td>298</td>
<td>24.331</td>
<td>5.70 \times 10^{-3}</td>
<td>3.377</td>
<td>0.99</td>
</tr>
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<td></td>
<td>10:1</td>
<td>308</td>
<td>30.864</td>
<td>5.85 \times 10^{-3}</td>
<td>5.577</td>
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<tr>
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<td>318</td>
<td>35.971</td>
<td>5.96 \times 10^{-3}</td>
<td>7.722</td>
<td>0.99</td>
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<tr>
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<td>15:1</td>
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<td>15.847</td>
<td>6.06 \times 10^{-3}</td>
<td>1.520</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
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<td>17.668</td>
<td>7.08 \times 10^{-3}</td>
<td>2.211</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>15:1</td>
<td>308</td>
<td>21.739</td>
<td>8.45 \times 10^{-3}</td>
<td>3.995</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>15:1</td>
<td>318</td>
<td>24.213</td>
<td>12.19 \times 10^{-3}</td>
<td>7.148</td>
<td>0.99</td>
</tr>
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</table>
is a Van't Hoff relation and the plot of ln$K_e$ vs. $1/T$ is used to find the values of $\Delta H^o$ and $\Delta S^o$ from the slope and the intercept. $\Delta G^o$ and $\Delta H^o$ are in J/mol, $\Delta S^o$ is in J/mol K.

The values of $K_e$, $\Delta G^o$, $\Delta H^o$ and $\Delta S^o$ for the extraction of cottonseed oil using n-hexane and ethanol as solvents were calculated using Eqs. (7) and (10) are given in Tables 2 and 3. The values of $\Delta G^o$ calculated were found negative for the oil extraction from cottonseed at all temperatures for both solvents and decreased from $-11531.86$ to $-18556.50$ J/mol for n-hexane and $-10419.74$ to $-20962.73$ J/mol for ethanol. The negative value for the Gibbs free energy for oil extraction from cottonseed confirm that this process is feasible and spontaneous and that the degree of spontaneity of the extraction increases with increasing temperatures as $\Delta G^o$ becomes more negative. The decrease in the negative value of $\Delta G^o$ with an increase in temperature indicates that the oil extraction from cottonseed becomes more favorable at higher temperatures. The values of $\Delta H^o$ calculated from the plot of ln$K_e$ versus $1/T$ were in the range of $43212.84$ to $57330.85$ J/mol and $84511.81$ to $85758.91$ for n-hexane and ethanol, respectively. The value of $\Delta H^o$ was positive, indicating that the extraction is endothermic and requires energy during the process. The positive value of entropy indicates that the process is irreversible.

2.7. Properties of oil extracted by the two solvents

The oil extracted by n-hexane and ethanol was tested for various parameters and the results are reported in Table 4. It may be seen that there is not much variation between the quality of the oil extracted by n-hexane or ethanol except that the color of the oil extracted by ethanol is slightly darker than the oil extracted by n-Hexane. This indicates that ethanol is a good solvent for oil. Acid value,
which is the indicator of free fatty acids present in the oil, is slightly higher in the oil extracted by ethanol. Iodine value, which is the indication of the unsaturation saturation level of the oil, is nearly the same in both the oils and saponification value, which indicates the average molecular weight of triglycerides of the oil, is also comparable. In a nutshell, the quality of the oil extracted by ethanol is comparable to the quality of the oil extracted by n-hexane except for the gossypol content, which can be practically completely removed during the refining process of the oil before actual consumption.

3. CONCLUSION

The study concludes that ethanol can be considered as an alternative solvent to n-hexane for oil extraction at the SR of 10:1 and 318K where the efficiency of extraction is comparable to that of n-hexane. The quality of oil after refining is also acceptable for food preparations. The extraction data are well described by second order kinetics. The initial rate of extraction is not appreciably affected by the SR but is largely affected by temperature. The positive value of $\Delta H$ indicates that the extraction is endothermic and requires an input of energy during the extraction process. The positive value of entropy indicates that the extraction process is irreversible. The negative value of the Gibbs free energy for the oil extraction process is comparable to that at 318K where the quality of the oil extracted is considered as an alternative solvent to n-hexane.

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Table 4
Comparison of quality parameters of oil extracted by n-hexane and ethanol

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Parameters</th>
<th>n-Hexane</th>
<th>Ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Color</td>
<td>Brown</td>
<td>Dark Brown</td>
</tr>
<tr>
<td>2</td>
<td>Specific gravity g/L</td>
<td>0.9182</td>
<td>0.9202</td>
</tr>
<tr>
<td>3</td>
<td>Refractive Index at 313K</td>
<td>1.4641</td>
<td>1.4647</td>
</tr>
<tr>
<td>4</td>
<td>Acid value</td>
<td>0.37</td>
<td>0.52</td>
</tr>
<tr>
<td>5</td>
<td>Iodine value</td>
<td>106.8</td>
<td>107.2</td>
</tr>
<tr>
<td>6</td>
<td>Saponification Value</td>
<td>194.5</td>
<td>197.8</td>
</tr>
<tr>
<td>7</td>
<td>Gossypol (%)</td>
<td>0.27</td>
<td>0.63</td>
</tr>
</tbody>
</table>

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