EXTRACTION OF JOJOBA OIL USING VARIOUS CONCENTRATIONS OF TWO DIFFERENT SOLVENTS

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ABSTRACT

The aim of this study was extraction of jojoba oil using different solvents. A mixture of water-hexane and water-ethanol are used as solvents to extract jojoba oil in a batch extraction process and compared with a pure solvent extraction process. The effects of particle size of crushed seeds, solvent-to-water ratio and time on jojoba oil extraction were investigated. The best recovery of oil was obtained at the boiling temperature of the solvent and four hour of extraction time. When seed particle size was 0.45 mm and a pure ethanol was used (45% yield of oil extraction), whereas, it was 40% yield of oil at 25% water- hexane mixture. It was revealed that the water-ethanol and water-hexane mixtures have an effect on the oil extraction yield. The particle size, had a great effect on the maximum oil extracted yields. The maximum yield of oil extraction using pure hexane (35%), while for pure ethanol, the maximum yield of the oil extraction (45%) for the solvent, by using the water-solvent mixture this percentage has significant increasing in water-hexane solvent system.

Keywords: hexane mixture, ethanol mixture, boiling temperature, Soxhlet extractor

استخلاص زيت الجوجوبا باستعمال تراكيز مختلفة من مذيبين مختلفين

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المستخلص

في هذه الدراسه , تم استعمال خليط من الماء والهكسان والماء والايثانول كمذيب لاستخلاص الزيت من بذور الجوجوبا وتمت المقارنه مع استعمال مذيب نقي من الهكسان والايثانول. تمت دراسه تاثير حجم الحبيبات وتاثير نسبه المذيب الى الماء و تاثير الزمن على استخلاص الزيت من بذور الجوجوبا. ان افضل استخلاص للزيت عند درجه غليان المذيب ولفتره اربع ساعات عندما كان حجم الحبيبات يساوي 0.45 ملم واستخدام كحول اثيلي نقي حيث كانت نسبه الانتاج 45%في حين كان الانتاج 40% عندما اصبح المذيب يحتوي على ماء بنسبه 25%. ان وجود الماء مع الايثانول اومع الهكسان له تاثير على عمليه استخلاص الزيت. ان حجم الحبيبات ايضا له تاثير كبير على استخلاص الزيت. ان اعلى انتاجيه تم الحصول عليها استعمال الهكسان النقي كانت35% في حين كانت اعلى انتاجيه عند استخدام الكحول النقي هي 45% , هذه النسبة لها زيادة كبيرة في نظام خليط الهكسان و الماء.

كلمات مفتاحية: خليط الهكسان, خليط الايثانول, بذور الجوجوبا , جهاز السوكسليت

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INTRODUCTION

Jojoba plant (*simmondsia chinensis*) has seeds of nut shaped form. It is around 1–2 cm long. Its color is red-brown to dark-brown (7). It was grown at different places around the world. It is now grown in countries at the Middle East it was adapted to water stress. In Iraq, it was planted at Al-Anbar province. It grows under severe environmental conditions (14). The seeds of jojoba plant contain a high percentage of oil ranged from 50% to 60% (8). This oil is odorless and colorless, the main contents are straight-chain mono-esters of C_{20} and C_{22} as alcohols and acids with the two double bond. The bonds are on each side of the ester bonds. The following formula represents an ester which is found in Jojoba oil (20):

$$CH_3(CH_2)_7CH=CH\text{-}(CH_2)_7CO\text{-}O$$
$$(CH_2)_{11}CH = CH(CH_2)_7CH_3$$

The almost triglycerides free oil makes this oil different from other seed oils. It can be considered a liquid wax and not a fat **(8), (21),** (22). The seeds with high oil content can be processed either by mechanical pressing followed by solvent extraction or by direct solvent extraction only using different solvents to obtain high oil percentage. The oil content of the final jojoba meal by the mechanical pressing process has a range of 20-24% for the first pressing process, and for the second press the oil extracted was found to be 13.7-18.4 wt. %. However, Wisniak (20) showed that only 35-42 wt. % of the oil was removed by the double pressing process, therefore, an oil extraction was advised to remove 9-10 wt. % of the remaining oil in the pre-pressed meal. When the oil from the pre-pressed meal is solvent extracted; the residual oil content in the meal can be reduced to a very small value of less than 1% (21), (22). Jojoba oil has been used in many industries because of its promising physical properties. These include high viscosity index in addition to high flash and fire points. It has a high dielectric constant, and high stability. The oil has low volatility. The repeated heating up to a temperature of 300°C has little effect on its properties. Under an atmosphere of nitrogen of 757 mm, it has a boiling point of 398 °C (4), (19). Sometimes the oil seeds need pretreatment steps or preparation. These include cleaning, hulling, crushing, flaking, cooking, and others which must be done prior to the extraction process. The previous researches have been investigated different solvents for oil extraction; benzene, alcohol, chloroform, carbon tetrachloride, benzene, heptane, hexane, isopropyl alcohol and tetrachloroethylene, etc (6), (12), (17). Hexane is most commonly used in the solvent extraction process. This is due to its relatively low toxicity and low cost (6), (12), (17). The performance of the solvents depends to a large extent on the chemical and physical properties. To choose a solvent, the primary desired end product must be considered; the oil or the meal. Some of the recent researches took other solvents into consideration **(9)**. Water and ethanol would be green alternative since they are having useful properties for a wide range of chemical processes, although, theses solvents have restrict yield of extraction. To replace a solvent, another one or a mixture of two solvents are used. This is done depending on the equivalent solvency (18). A maximum leaching efficiency of a solute is needed in choosing a particular solvent. The mass transfer during the extraction of oil from the plant seeds depending mainly on, intra-particle diffusion (transport of the oil component), external diffusion (diffusion of oil through an outside stagnant liquid film of the seed) and removal of the oil to the adjacent solvent (5). Furthermore, solvent extraction is affected by the penetration of the solvent into the solid and dissolving the solute into the solvent. Therefore, in order to increase the solute solubility, a high extraction temperature is desirable (23). Finally, the process of solvent recovery is an important process and it must be done with minimum losses so solvents can be reused again. There are many method to recovery the solvent, the most common method is a simple distillation followed by a vacuum distillation process. Sometimes, traces of the solvent remain in the extracted oil. This depends on the process of solvent recovery (1), (2) , (3). In this research, the extraction of jojoba oil was studied using different solvents with different concentrations and variable factors affecting the extraction process

MATERIALS AND METHODS

All seeds of jojoba were collected, washed to remove impurities, dried in 110ºC for 24 hours to get rid of water prior any further treating then a 10 grams of seed were crushed to have either coarse particles (1.5 mm) or fine particles (0.45mm). The extraction process using Soxhlet extractor shown in Figure 1 was done using two aqueous solvents; n-hexane (95% ALDRICH) and ethanol. The organic solvents, have different polarities ranging from highly non-polar solvent (hexane) to moderate polar solvent (ethanol), their specification as shown in table 1. Different concentrations of solvent-water solutions were used. These were: 75%, 50%, and 25% (Vol. %), in addition to the pure solvents. The weighed crushed seeds were mixed with 100 ml of the solvent. The extraction process was carried out at the boiling point of the solvent for a four hours0 Then, the oil was recovered using a rotary evaporator (RE3022C STUART) as shown in Figure 2 to separate the oil-solvent mixture so that almost complete removal of solvent was conducted. The extracted oil was estimated by using FTIR analysis (Genesys 10 UV) and by weight.

Table 1. physical properties of solvents.

Property	Hexane	Ethanol	Water
Chemical formula	C_6H_{14}	C_2H_6O	H_2O_2
M. Wt. (g / mol)	100.2	46.07	18
Density(g cm-3)	0.659	0.789	0.998
Boiling point (°C)	69	78.4	100
Solubility in water	Immiscible	Totally miscible	-
Polarity index	0.1	5.1	10.2

The yield of oil extraction was calculated according to the following equation:

$$\text{Yield} = \frac{\text{weight of oil extracted}}{\text{weight of crushed seeds}} \%$$

The next step is conducting the tests which included: FTIR to examine the ingredients of the samples.

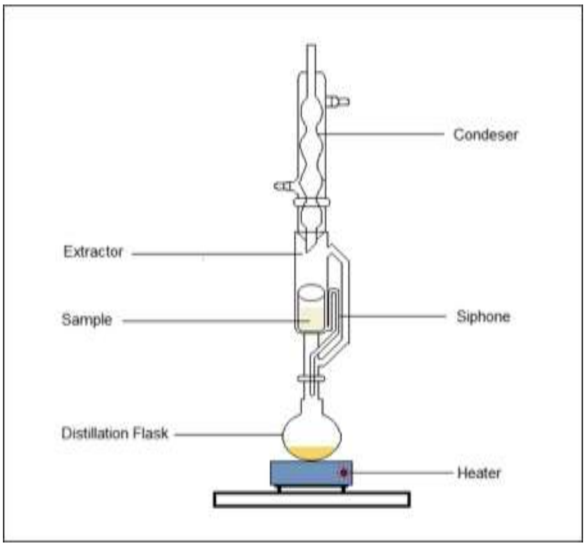

Figure 1. Extraction setup (Soxhlet extractor)

Figure 2 . Rotary evaporator

RESULTS AND DISCUSSION

The effects of particle size and solvent type on the extraction of jojoba seed oil were investigated in a bench scale system using Soxhlet extractor. Extraction yield was studied using a pure solvent (ethanol or hexane) and a water-solvent mixtures as alternatives to typical solvents. Although water is highly polar solvent it has extraction limitations to dissolve jojoba seed oil (non-polar substances of jojoba). Therefore, the solubility in a polar solvent (water) has been enhanced using different volume percentages of organic solvents to extract non-polar solute. Finally, at the best yield of oil extraction different particle sizes (coarse, and fine) of jojoba seeds have been studied to identify the effect of particle size of seeds on oil yield.

Effect of solvent

The effect of the pure solvent (ethanol or hexane) on the extraction of jojoba oil was studied by changing the time of extraction and the particle size of the seed, Fig. 3 and Fig. 4. It can be seen that for both ethanol (Fig. 3) and hexane (Fig. 4) a reduction in particle diameter from 1.5 mm to 0.45mm had been reflected positively on the yield of oil extraction. The initial yield of oil extraction after 30 min in Fig.3 was increased by about 10%. By prolonging the period of extraction, the yield of oil extraction for the particle diameter of 1.5 mm and 0.45 mm reached a closer value. However, when particle size was reduced, the internal resistance of mass became smaller, and this has a positive influence on increasing the initial and final yield of oil extraction. Fig 4. shows the same behavior as in Fig. 3. The yield of oil extracted was initially increased by about 9% by reducing particle diameter from 1.5 mm to 0.45 mm. The yield of oil extraction at this period is fast due to the leaching of accessible solute from the destroyed cell walls. Then, the rate of oil yield extracted using ethanol-water system was increased slightly after the four hours of leaching, and in order for comparing between the water-ethanol system and water-hexane system the four hours leaching time have been at same operation conditions have been chosen (16). From these results, it was concluded that the yield of oil extraction could be enhanced; by reduction of the particle size. Also, the yield of oil extracted using ethanol was higher than the yield using hexane under the same conditions.

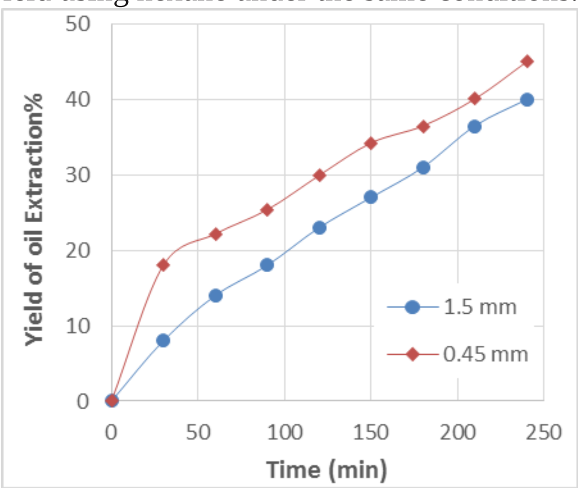

Figure 3. Extraction yield percentage rate of ethanol.

Finally, in order to obtain a maximum yield of extraction, the internal resistance has to be reduced by destroying the jojoba cellular structure.

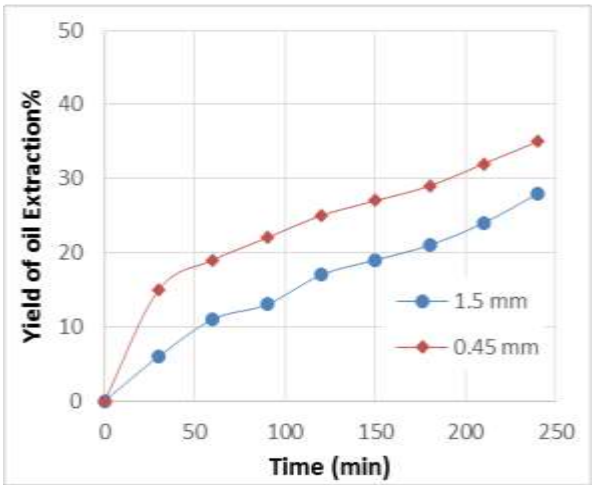

Figure 4. Extraction yield percentage of hexane

Effect of the water- solvent mixture

The effect of water-solvents mixture (0, 25, 75, 100 %) on the extraction yield at different their solvents points for the 0.45 mm size of jojoba seed, and 4 h extraction time are shown in Fig. 5. The results showed that water-ethanol mixture had higher efficiency during 4 hours of extraction compared to the water-hexane mixture. This behavior can be explained due to the time of extraction (4 hours). It is clear that the water-ethanol mixture needed less time to extract the jojoba oil. Most researches like (8) , (9) and (10) showed the extraction time by hexane using Soxhlet was between 8 to18 hours. However, in this research, the time of extraction was fixed four hours for all experiments to make comparison between solvents.

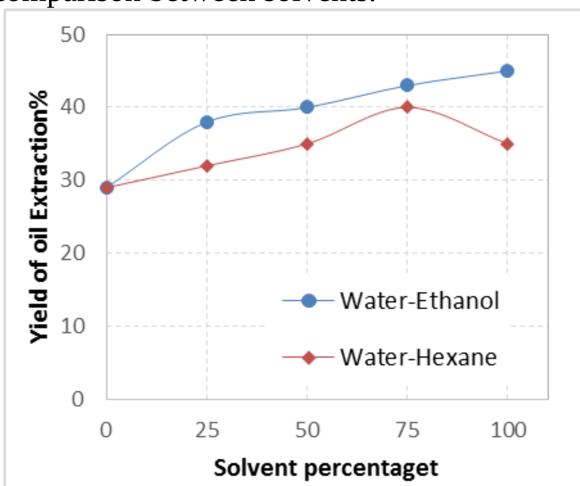

Figure 5. Effect of water-solvents ratio on extraction yield

Fig 5. demonstrate that oil extraction yield was 29, 38, 40, 43 and 45 % for the water-hexane mixture, while for the water-ethanol mixture the yield was 29, 32, 35,40 and 35%. These represented a water content of 100, 75, 50, 25 and 0 % respectively in the mixture. The oil extraction yield of 45% for pure ethanol was the highest value for time of 4 h, whereas the oil extraction yield for the water-hexane mixture gave the highest value with 25% water content which can be explained by increasing the polarity of the solvent mixture system. The oil extraction yield by water-ethanol mixture are more than those for water-ethanol mixtures because water-ethanol mixtures are more polar than hexane-water mixtures they had so more tendency to dissolve polar substances in jojoba oil. At lower ethanol concentrations, jojoba oil solubility was reduced. Water control in alcohols mixture becomes essential in order to preserve oil solubility (15). In addition, the 40% oil extraction yield of 75% hexane solutions was better than the value of oil extraction yield for pure hexane at the same extraction period. That might be due to theslightly increasing in polarity of the mixture and that is in agood agreement with (13). Megan reported an increasing in extraction capacity of polar substance in soybean extraction by increasing the water content of alcohol aqueous solution (13). This means the increase dissolution of the polar substances in the jojoba seed. Fig. 6 and Fig.7 show the effect of the jojoba seed particle size on the oil extraction yield after 4 hours of extraction time at the mixture boiling points.

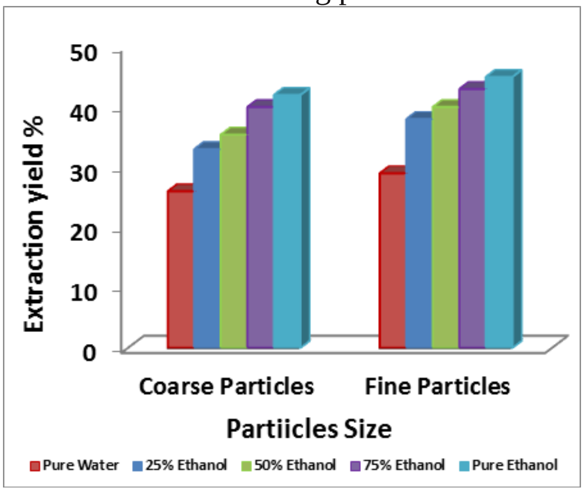

Figure 6 . Effect of particle size on extraction yields with different water-ethanol mixture

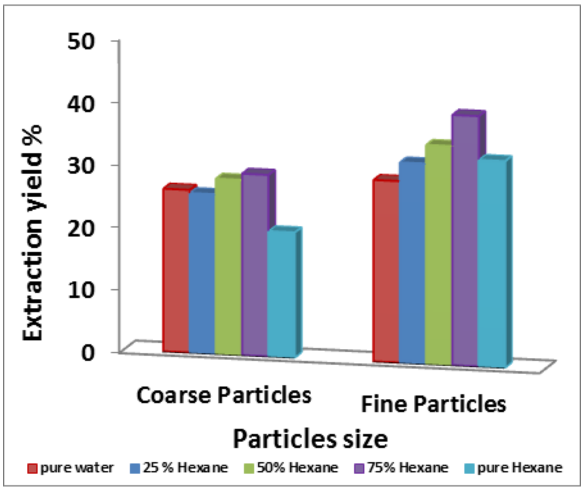

Figure 7. Effect of particle size on extraction yields with different water-hexane mixture

For all mixtures, reduction in particle size from 1.5 mm to 0.45 mm increased the oil extraction yield and extraction process was enhanced. Crashing seed particles leads to breaking the internal seed structure that allowed the solvent to be diffused inside the particles and leach the solute. Also the increase of the oil extraction amount with reduction in particle size may be explained through the increasing of oilseed materials (16). For water-hexane mixtures, the oil extraction yield was increased for the same period (Fig. 7). It was clear that when the water content was 25 % and hexane was 75% of the solvent mixture, the oil extraction yield percentage reached up to 40% for the same period. The increase in oil extraction yield percentage was significant which confirms the effect of the solvent polarity in addition to the particles diameter for the extraction process , these results ties well with previous studies(13), (16). From Fig.6 Fig.7, it was conclud that by reduction of the particle size of the seed, the extraction process is enhanced and high oil extraction yield was obtained during a shorter time. Therefore, reducing internal resistance assisted in faster diffusion of solvent into the small particles which led to diffusion of the solute from the particles and higher oil extraction yield.

FTIR spectroscopy

The FTIR spectrum of pressed jojoba oil was compared with FTIR spectrum of collected extracted jojoba oil samples. Fig. 8 , Figure 9 and Figure 10 shows similarity of FTIR spectrum for pressed jojoba oil with jojoba oil extracted using pure solvents, the broadband between 2852- 3003 cm^{-1} characterize the jojoba oil. Fig. 9 illustrates nearly the disappearance of OH^- stretching vibration at $3342cm^{-1}$ and that means the oil is in a pure state, meanwhile, the wide broadband appeared at 3342 cm^{-1} in Fig. 10 using hexane as a solvent. The spectra for pressed and extracted jojoba oil have the identified C=O band at 1739 cm^{-1} and at $1461cm^{-1}$ due to the double bond. All samples showed the presence of one or two double bonds. This finding is in good similarity with the standard jojoba oil analysis.

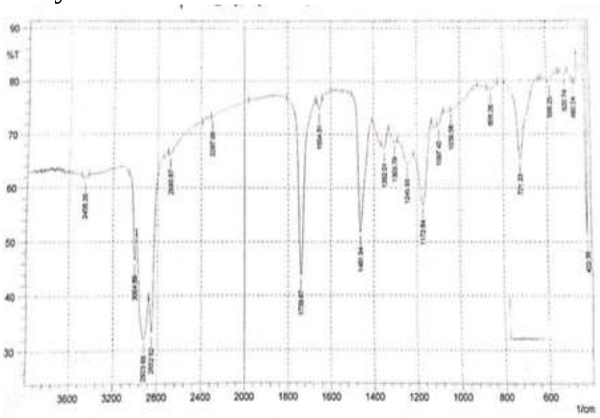

Figure 8. FTIR spectra of pressed jojoba oil

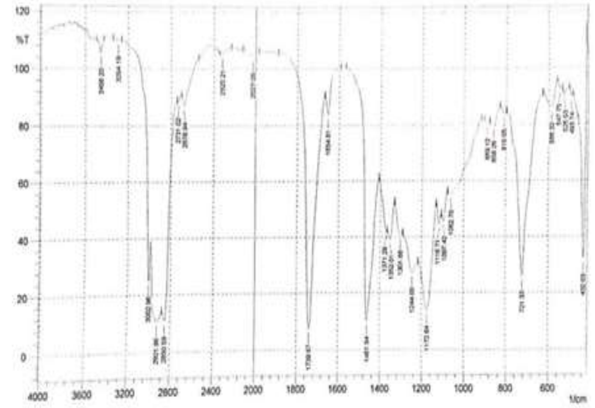

Figure 9. FTIR spectra of jojoba oil extracted using ethanol

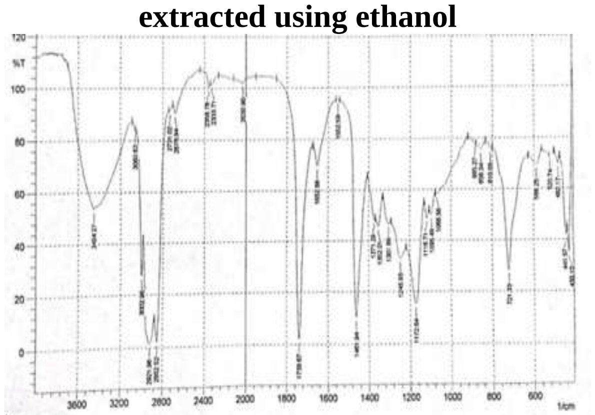

Figure 10. FTIR spectra of jojoba oil extracted using hexane

From these results of this research, it can be conclude that all the water–solvent mixtures have effects on the oil extraction yield. The maximum oil extraction yields were obtained from the jojoba seeds by using good polar solvent and a smaller particle size. For pure hexane, the maximum yield of oil extraction was 35%, whereas, by adding 25% of water to hexane solvent the yield of oil extraction increased to 40%. For ethanol, the maximum yield of the oil extraction was 45% for pure solvent. It was clear that for the reduction in particle diameter from 1.5 mm to 0.45 mm reflected positively on the yield of oil extraction

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