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Leaching of Jojoba Oil

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Abstract

The leaching process was applied to extract jojoba oil by means of Multifunctional Extraction Plant which presents in Specialized Training Institute for Chemical Industries (STICI).Three solvents were examined; n-hexane, diethyl ether, and petroleum ether.The purpose of work is to select the best solvent which extracts more oil and comparing among them according to cost and energy consumption during the leaching operation.The results of our study indicate that the best solvent was n-hexane; which the jojoba oil leached using this solvent was the largest amount. The n-hexane also the less one has consumed the energy.The other purpose is to study the relationship between seeds' particle size and the oil yield; therefore, two different particle sizes of jojoba seeds were employed. From the smaller one we get highest oil yield.

Keywords: Jojoba oil, n-hexane, Diethyl ether, Petroleum ether, Leaching

1- INTRODUCTION

1.1 Jojoba Shrubs

Jojoba (pronounced ho-ho-ba) is a desert shrub that grows wild in southern Arizona, north-western Mexico and neighboring areas, recently it has been planted in middle-eastern countries. Jojoba seed contains a light-gold colored liquid wax ester which is the primary storage lipid of the plant. This is unlike conventional oilseed crops, such as soybean, corn, olive, or peanut which produce oils as the primary storage lipid. Jojoba wax (called oil) contains about 50 % of the seed's dry weight. The physical properties of jojoba oil are: high viscosity, high flash and fire point, high dielectric constant, high stability and low volatility. Its composition is little affected by temperatures up to 570 °F (300 °C). Jojoba oil contains straight-chained C20 and C22 fatty acids and alcohols and two unsaturated bonds, which make the oil susceptible to many different types of chemical manipulations. The extracted oil is relatively pure, non-toxic, biodegradable, and resistant to rancidity.

Interest in jojoba oil stems from its unusual properties which differs from all known seed oils. The complete absence of glycerin makes it a liquid wax, not a fat. Jojoba oil has been evaluated for suitability in many applications such as cosmetics, pharmaceuticals, lubricants, food, electrical insulators, foam control agents, high- pressure lubricants, heating oil, plasticezers, fire retardents, and transformer oils, plus others. Jojoba is new in Jordan: In 1986, the first jojoba plants were planted at the Jordan University of Science and Technology (J.U.S.T). The jojoba seeds used in this study were taken from this farm.

Jojoba is a woody evergreen shrub or small multi-stemmed tree that typically grows to a height of 10 to 15 ft. Leaves are opposite, oval or lanceolate, gray green, and have a waxy cuticle that reduces moisture loss. The plant develops one or a few long tap roots (up to 40 ft) that can supply water and minerals from far below the soil surface. Figure (1.1) shows a jojoba plant.

Figure (1.1) Jojoba shrub

Jojoba is usually dioecious (male and female flowers are borne on separate plants). Female flowers are small, pale green and commonly solitary or in clusters at the nodes. Male flowers are yellow, larger, and occur in clusters. Pollination occurs via wind or insect [1]. The fruit is a green capsule which encloses up to three seeds. When ripe (3 to 6 months after fertilization) the capsule splits and reveals the seed, which is brown, wrinkled and about the size of a small olive (300 to 1,000 seeds/lb). Seed production is

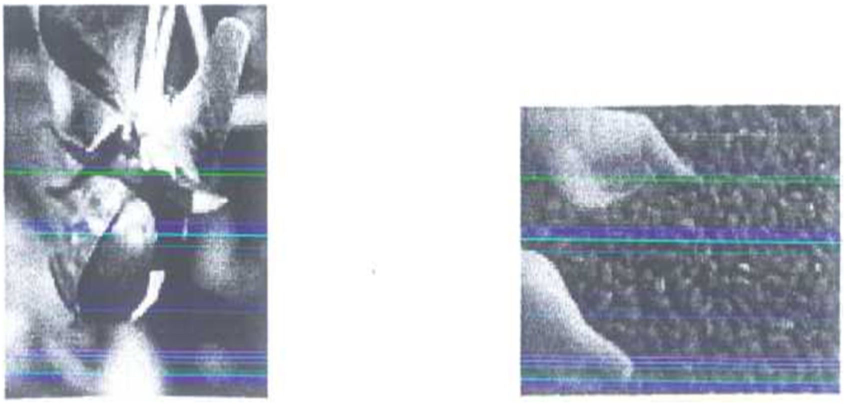

Figure **(1.2):** a) **Jojoba** fruit, **b) Jojoba seeds**

Jojoba is a native to the Sonoran Desert. Most natural populations exist only inside a quadrangle with Riverside (California), Globe (Arizona), Guaymas (Sonora, Mexico), and Cabo San Lucas (Baja California, Mexico) as it's four comers, and comprise about 100,000 square miles between latitudes 25° and 34° North. These disjunctive jojoba populations occur from sea level to about 1,500 m altitude on coarse, -sandy or gravelly soils with good water drainage. The rainfall in the area ranges from 75 mm to about 400 mm, and the plant communities are exposed to temperatures between -9°C and 50°C.

Jojoba is a perennial, dioecious, evergreen shmb or small tree that lives under diverse environmental conditions. It has an extensive and deep root system and requires little care if maximum seed production is not desired. Jojoba is valuable as a soil conservation and landscape plant for highway shoulders, city parks, and other places that cannot afford much care [1].

Irrigation has produced more luxuriant vegetative growth, but it is not known whether this increased growth results in higher seed yield. Jojoba requires the most water during late winter and early spring. This desert plant is extremely drought tolerant, and is well suited to a broad area of inland Jordan where it offers not only stable production, but environmental benefits not offered by existing land-use practices. These will ameliorate some of our land degradation problems.

Most wild jojoba populations occur on coarse, light or medium textured soils with good drainage and good water infiltration. Planting on heavy soil results in later blooming, slower growth and more problems with fungal diseases. Jojoba can be planted by direct seeding or by transplanting seedlings to the field. In the southwestern U.S. many growers prefer direct seeding because it is less expensive, faster and requires less hand labor. Seed can be germinated in vermiculite or sand at about 80 °F. Emergence occurs in 15 to 20 days, and the seedlings are ready for transplanting when they are 6 to 12 in. tall (8 to 10 weeks). Emergence from direct-seeded fields occurs in 15 to 20 days. Propagation from clones or from tissue culture is a more rapid method of varietal improvement.

1.2 Jojoba Oil

Interest in Jojoba stems from the unusual properties of the oil that can be extracted from its seeds. Most seeds contains between 45 to 60 % oil by weight, more than twice the amount found in soybeans and somewhat more than in most oilseed crops.

The Jojoba oil is odorless, colorless oil and is composed principally of 40 and 42 carbon chain length esters of the straight chain mono-esters of the 20 to 22 carbon atoms alcohols and acids, with two double bonds, one at each side of the ester bond (as shown in figure (1.3)). The almost complete absence of glycerin indicates that jojoba differs radically from all known seed oil. It is not a fat, but a liquid wax.

m=713 , n =814

Figure (1.3) Jojoba chemical structure

Jojoba oil is easy to work with, it is nontoxic and biodegradable. It dissolves readily in common organic solvents such as benzene, petroleum ether, chloroform, carbon tetrachloride, and carbon disufilde, but it is immiscible with methanol and acetone.

The nature of the oil can be radically changed by reactions at the double bonds and ester functions.

Many new products can result (more than 40 different jojoba- based chemicals) and appear to have commercial industrial applications. As with other natural oils, the double bonds in fresh Jojoba oil are all in the "cis" configuration. However, they can be easily isomerized (twisted around in space) using as catalysts traces of selenium, nitrogen oxides, or active earth. This produces an equilibrium mixture with 20% "cis" and 30% "trans" double bonds. A by-product of jojoba nuts is the meal filler cake remaining after the oil has been extracted. This material constitutes about 50% of the nuts, so that any commercial operation will have to consider the handling of large amount of meal and its potential uses. This is an area that so far has not received enough attention; its solution would improve the economic attractiveness of the industry.

The present experience gives some basic information on the composition of the meal, as well as its ammo acid content. Of the essential amino acids, the lysine content -is_good, but the methionine content is poor. The protein content varies between 26% and 33% making the meal attractive as a livestock feed. The meal may also be mixed with dry nonfat milk solids (75% meal to 25% milk) to use as a pollen substitute for bees [2].

1.3 physical Properties

For many industrial needs, the oil has promising physical properties : high viscosity index, high flash and fire points, high dielectric constant, and high stability. It has low volatility and its compositions little affected by repeated heating to remarkably high temperature up to 300 °C. Its boiling point at 757 mm under nitrogen is 398 °C.

Jojoba oil also has good keeping, qualities and an exceptional shelf life. This is apparently due to the presence of natural antioxidants (alpha-, gamma-, and delta- tocoperols), which occur in concentrations of about 50 ppm. In practical terms, these antioxidants keep the oil from becoming rancid. Companies processing raw jojoba oil are reporting very low acid values (0.2 to 0.3), even without neutralization [2]. The physicochemical properties of Jordanian jojoba oil is determined in previous work, and is listed in table (1.1).

1.4 Usage of Jojoba Oil

The cosmetic industry appears to be the principal market for jojoba oil products, around 2,000 tons per year are thought to be utilized by this industry, this equates to almost 80 % of the total market share. The other major industry using jojoba oil is the pharmaceutical sector. Lubricant applications provide a market for around 100 tons of jojoba oil annually.

Jojoba oil is a high quality substitute for sperm oil (from the sperm whale), availability if this product is limited now due to the restrictions placed on whaling. The meal left over from crushing has high protein content but needs detoxification before it can be fed to livest.

The shrub’s peanut-sized seeds produce an oil which engineers at the United Arab Emirates University in Al-Ain and the Helvvan University in Cairo combined with methanol to make a fuel called " jojoba methyl ester ", which was successfully tested as a fuel for vehicles. Table (1.2) gives potential uses of jojoba oil.

Property	Its value
Specific gravity at 25 °C	0.861
Plash point (open cup) (°C)	275
Flash point (closed cup) (°C)	244
Fire point (°C)	322
Pour point (°C)	8
Aniline point (°C)	52.9
Carbon residue (%wt)	0.012
Iodine value (g per 100 g)	81
Saponification value *	88
PH	6.95-7.34
Calorific value (cal/g)	10086
Refractive index at 25 (°C)	1.4593
Moisture content by infrared (%Water)	0.64
Foaming at 24 & 94 °C (ml)	3-5
Color value	1.0
Ash content (%wt)	0.0
Viscosity (cSt) at 40 °C at 100 °C	24.75 6.43
Viscosity index	233
Cu-corrosion	< 1 a

Physicochemical properties of Jordanian Jo joboba oil [3]

Table (1.2): potential uses of jojoba oil [4]

Type of Modification	Description of product	Proposed Uses
Filtered natural oil	Clear liquid, odorless, stable, indigestible	1. Dietary cooking oil 2. Cosmetics 3. Anti-foam agent in antibiotic fermentation 4. Leather fat-liquoring 5. Blending with polyethylene as a plastic modifier
Isomerization	Thick opaque cream	1. Cosmetic creams 2. Pharmaceutical creams
Hydrogenation *	Hard sparkling white semi-crystalline wax	1. Candles, polishes 2. Treatment of papers, textiles, insulating materials, matches, carbon paper 3. Manufacture of soap, salves, crayons etc 4. Coatings for foodstuffs
Treatment with sulfur chloride	A range of forms from oils to rubbery solids (factices)	1. Manufacture of varnishes, rubber, adhesives, linoleum 2. Printers ink
Sulfurization	Liquid	1. High pressure, high temperature lubricants for use in gearboxes, differentials etc 2. Crankcase lubricant 3. Cutting oils for use in machine shops 4. Lubricant for videotapes

1.5 Why has been Jojoba OH Utilized intensively in Cosmetics Industry?

Olive oil, beeswax, and fats are traditional cosmetics bases, but jojoba has important aesthetic and technical qualities that could also make it a widespread basic cosmetics ingredient. Indeed, in recent trials, cosmetics researchers have found that refined jojoba oil was-superior to all alternatives. It has no resins, tars, glycerides, alkaloids, glucosides, or low-molecular-weight fatty acids. And, because of its stability toward rancidity and its pleasant feel on the skin, it could become a standard oil-phase base for the cosmetics industry. Figure (1.4) illustrates the jojoba oil penetrating skin.

Jojoba oil has already been widely used in cosmetics in the United States and Europe. As many as 300 products have appeared in United States markets alone. But most of these have used jojoba oil only for its novelty value, not for its fundamental qualities. Such health and beauty care products have been successful largely because jojoba oil is easily substituted for other oils. But manufacturers are now coming to realize that the oil has high aesthetic qualities, better stability than even mineral oil, and lacks the problems of conventional triglyceride oils.

Among the varied products already being sold arc shampoos, hair conditioners, hair sprays, facial oils, body oils, bath oils, hand lotions, moisturizers, simian lotions, make-up removers, shaving creams, lipsticks and lip glosses, vanishing creams, cleansing creams, and skin fresheners [5]

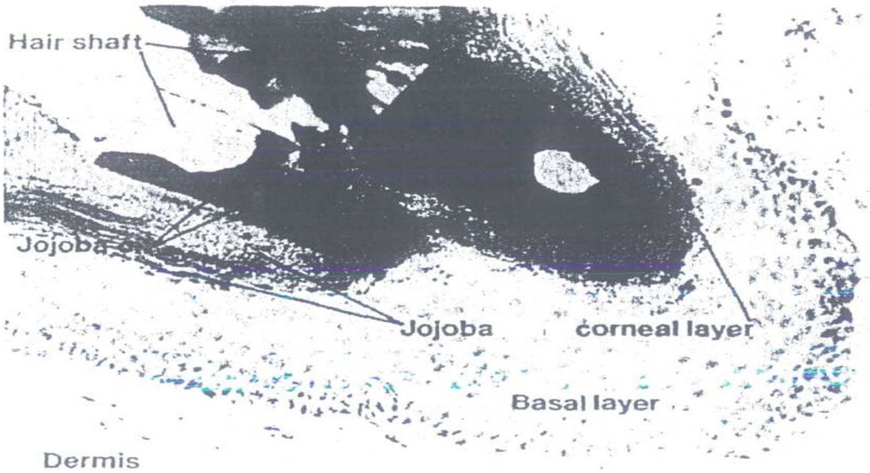

Figure (1.4): Jojoba oil penetrating skin. Experiments at the University of Michigan show that jojoba oil has an apparently unique ability to penetrate skin. Here, special slums show the oil pooled at the bottom of a hair and moving through the follicle wall into the corneal layer. This demonstrates jojoba oil's potential 10 soften skin from within, a property that could lead to it becoming a basic ingredient in many cosmetic products. (K. D.

McClatchy)

1.6 Extraction of Joioba Oil

Jojoba oil can be recovered either by pressing and/or leaching using different solvents. Previous work has concentrated on jojoba oil recovery using pressing and leaching. To achieve a high percentage of oil recovery (about 34%) using pressing, a Mpa pressure is required. Not all the oil is recovered which means a second pressing or leaching is required for the jojoba seeds. Continuous leaching process has been studied where a specific solvent has been used. Static or batch leaching has not been studied to examine other parameters that may affect oil recovery.

Leaching process using solvents may be used lo extract the oil mainly if it is going to be used in chemical industries and not for human consumption or uses. Traces of solvent may remain in oil depending on the solvent recovery process. Solvent can be recovered with minimum losses to be reused again. The process of solvent recovery could be a simple distillation followed by a vacuum distillation process [3].

1.7 Project Objectives

The objectives of this project are:

1. To study the feasibility of leaching jojoba by batch wise process using Multifunctional Extraction Plant.
2. To determine a most suitable solvent to be used in process.
3. 3. To study the effect of crushed jojoba seeds' diameter on the oil yield.

2. THEORETICAL BACKGROUND

2.1 Leaching Process

Leaching is the removal of a soluble fraction, in the form of a solution, from an insoluble, permeable solid phase with which it is associated.

The separation usually involves selective dissolution, with or without diffusion, but in the extreme case of simple washing it consists merely of the displacement (with some mixing) of one interstitial liquid by another with which it is miscible. The soluble constituent may be solid or liquid; and it may be incorporated within, chemically combined with, adsorbed upon, or held mechanically in the pore structure of the insoluble material. The insoluble solid may be massive and porous; more often it is particulate, and, the particles may be openly porous, cellular with selectively permeable cell walls, or surface-activated.

Because of its variety of applications and its importance to several ancient industries, leaching is known by a number of other names. Among those encountered in chemical engineering practice are extraction, solid-liquid extraction, lixiviation, ′ percolation, infusion, washing, and decantation-settling. The stream of solids being leached and the accompanying liquid is known as the underflow; in hydrometallurgy practice it is called pulp. The solid content of the stream is sometimes called marc (particularly by oil seed processors). The stream of liquid containing the leached solute is the overflow. As it leaves the leaching process it has several optional names: extract, solution, lixiviate, leachate, or **miscella [6].**

2.2 Leaching Process Mechanism

In the leaching of soluble materials from inside a particle by a solvent, the following general steps can occur in the overall process: The solvent must be transferred from the bulk solution to the surface of the solid. Next, the solvent must penetrate or diffuse into the solid. The solute dissolves into the solvent. The solute then diffuses through the solid-solvent mixture to the surface of the particle. Finally, the solute is transferred to the bulk solution. The presence of many different phenomena makes it almost impracticable or impossible to apply any one theory to the leaching action.

In general, the rate of transfer of the solvent from the bulk solution to the solid surface is quiet rapid, and the rate of transfer of the solvent into the solid can be some what rapid or slow. These are not, in many cases, the rate limiting steps in the overall leaching process. Solvent transfer usually occurs initially when the particles are first contacted with the solvent. The dissolving of the solute into the solvent inside the solid may be simply a physical dissolution process or an actual chemical reaction that frees the solute for dissolution. In case of vegetable oils, the oil "solute" dissolves into the solvent by a physical dissolution, rather than a chemical reaction. The rate of diffusion of the solute through the solid and solvent to the surface of the solid is often the controlling resistance in the overall leaching process and can depend on a number of different factors. If the solid is made up of an inert porous solid structure with the solute and the solvent of the pores, the diffusion through the porous solid can be described by an effective diffusivity. The void fraction and trotuosity are needed. In biological or natural substances additional complexity occurs because of the cells presence.

2.3 Variables Affect the Leaching Process [21]

The leaching process is influenced by many parameters, such as a temperature of the process, type of solvent, particle size of seeds, mixing speed of solid-liquid mixture, and initial solvent concentration per solid weight. In our work; not all the previous variables will be studied, because they are fixed variables by the design of the operating unit Only the effect of the particle size and type of the solvent on the batch leaching process will be presented.

2.3.1 Temperature Influence

It is usually desirable to leach at as high temperature as possible. Since higher temperatures results in higher solubility of the solute in the solvent, higher ultimate concentration in the leach liquor are possible. Viscosity of the liquids decreases and diffusivities increases with temperature leading to increased rates of leaching [16], So, the temperature has a positive influence on the leaching process. The temperature used in our work in fixed at the boiling point of the solvent.

2.3.2 Effect of seed particle diameter

Solid feed preparation usually plays a crucial role in leaching processes. The use of line particles usually will speed up diffusion transfer of solutes from the solid lo the extract.

Vegetable and animal bodies are cellular in structure, and the natural products to be leached from these materials are usually found inside the cells. If the cell walls remain intact upon exposure to suitable solvent, the leaching action involves osmotic passage of the solute through the cell walls. This may be slow, but it is impractical and sometimes undesirable to grind the material small enough to release the contents of individual cells. Thus, to increase the surface area of particles, vegetable seeds are usually rolled or flaked to give particles in the size range 0.15 to 0.5 mm before leaching in order to reduce the time required for the solvent to reach the individual plant cells. So, smaller particle diameter of seeds leads to a larger oil yield in appropriate time, which results saving energy.

2.3.3 Mixing Speed Influence

As we seen from the leaching process mechanism, there are two resistances, i.e. internal and external resistances. The internal resistance which is due to diffusion process can be reduced by reducing the particle size of seeds, While external resistance takes place in solution, so, ensuring good mixing would reduce this resistance significantly (keeping uniform distribution of solute concentration in the solution). So, the mixing process is a function of speed up the leaching process by decreasing the external resistance (Bulk resistance). Unfortunately, the apparatus we employed does not have a mixer.

2.3.4 Effect of Solvent-to-Seeds Ratio

Mass transfer process occurs due to a concentration gradient. So, when the solute concentration in the bulk is less than its concentration inside the solid, the diffusion process continue until the concentrations of solute in the bulk and inside the solid are in equilibrium. Therefore, when the solvent amount is increased relative to seed weight, the leaching role will increase. Increasing the solvent-to-seeds ratio will increase the oil yield up to a point where increasing the amount of solvent will not have an effect on leaching rate. In our work the solvent-to-seeds ratio varies with time, and—reaches the maximum value of 28 ml/g before starting the solvent circulation.

2.4 Organic Solvent Extraction

Many naturally occurring organic products are separated from its original structure by leaching. For example, vegetable oils are recovered from seeds such as soybeans and cottonseed by leaching with organic solvent, tannin is dissolved out of various tree barks by leaching with water, and many pharmaceutical products are similarly recovered from plant roots and leaves.

All vegetable seeds are regularly leached, or extracted, with solvent for removing the vegetable oils they contain. The seeds must usually be specially prepared for most advantageous leaching, and this may involve dehulling, precooking, adjustment of the moisture (water) content, and rolling or flaking. Leaching solvents are usually petroleum naphtha's. For most oils a fraction corresponding closely to hexane, and chlorinated hydrocarbons make the leached meal too toxic to be used as an animals feed. The oil-solvent solution, which usually contains a small amount of finely divided, suspended solids, is called "miscella" and the leached solid "marc" [2].

2.4.1. Solvent Selection

The leaching process depends on the chemical structure of the solvent and the kind of solute that will be extracted from solid materials. In many cases the presence of similar function groups in the molecules suffices, when a chemical similarity is present. The solution of the two components will usually have a structure similar to that of the pure material. This rule of thumb has only limited validity since there are many examples of solutions of chemically dissimilar compounds. On the other hand, there is a wide range of possibilities where the two materials dissolve each other to a limited extent.

A solvent should not be considered a macroscopic continuum characterized only by physical constants such as density, dielectric constant, index of refraction....etc.., but as a discontinuum which consists of individual, mutually interaction solvent molecules. According to the extent of these interactions, there are solvents with a pronounced internal structure such as water, and others in which the interaction between the solvent molecules is small such as hydrocarbons. Rather than the “Like dissolves like" rule, it is the intermolecular interactions, between solvent and solute molecules, which determines the mutual solubility.A compound A dissolve in a solvent B only when the intermolecular forces of attraction KAA and KBB for the pure compounds can be overcome by the forces K_{AB} in the solution. The sum of the interaction forces between the molecules of solvent and solute can be related lo the so-called polarity of A and B. Polar compounds have a - large interaction molecules forces, and non-polar have a small ones[3]

On the other hand, there is another factor that can be used in the estimation of solubility of non-electrolytes in organic solvents which called 5.

The solubility parameter is a numerical value that indicates the relative solvency behavior of a specific solvent. It is derived from the cohesive energy density of the solvent, which in turn is derived from the heat of vaporization. What this means will be clarified when we understand the relationship between vaporization, Van der Waals forces, and solubility.

When a liquid is heated to its boiling point, energy (in the form of heat) is added to the liquid, resulting in an increase in the temperature of the liquid. Once the liquid reaches its boiling point, however, the further addition of heat does not cause a further increase in temperature. The energy that is added is entirely used to separate the molecules of the liquid and boil them away into a gas. Only when the liquid has been completely vaporized will the temperature of the system again begin to rise. If we measure the *amount* of energy (in calories) that was added from the onset of boiling to the point when all the liquid has boiled away, we will have a direct indication of the amount of energy required to separate the liquid into a gas, and thus the amount of Van der Waals forces that held the molecules of the liquid together.

It is important to note that we are not interested here with the temperature at which the liquid begins to boil, but the amount of heat that has to be added to separate the molecules. A liquid with a low boiling point may require considerable energy to vaporize, while a liquid with a higher boiling point may vaporize quite readily, or vise versa. What is important is the energy required to vaporize the liquid, called the **heat of vaporization.** (Regardless of the temperature at which boiling begins, the liquid that vaporizes readily has less intermolecular stickiness than the liquid that requires considerable addition of heat to vaporize.)

From the heat of vaporization, in calories per cubic centimeter of liquid, we can derive the cohesive energy density (c) by the following expression (equation 2.1):

$$c = \frac{AH - RT}{Vm} \qquad \ldots \qquad (2.1)$$

here: c = Cohesive energy density , AH = Heat of vaporization , R = Gas constant, T = T emperature, V_m = Molar volume.

In other words, the cohesive energy density of a liquid is a numerical value that indicates the energy of vaporization in calories per cubic centimeter, and is a direct reflection of the degree of Van der Waals forces holding the molecules of the liquid together.

Interestingly, this correlation between vaporization and Van der Waals forces also translates into a correlation between vaporization and solubility behavior. This is because the same intermolecular attractive forces have to be overcome to vaporize a liquid as to dissolve it. This can be understood by considering what happens when two liquids are mixed: the molecules of each liquid are physically separated by the molecules of the other liquid, similar to the separations that happen during vaporization. The same intermolecular Van der Waals forces must be overcome in both cases.

Since the solubility of two materials is only possible when their intermolecular attractive forces are similar, one might also expect that materials with similar

cohesive energy density values would be miscible. This is in fact what happens.

In 1936 Joel H. Hildebrand (who laid the foundation for solubility theory in his classic work on the solubility of non-electrolytes in 1916) proposed the square root of the cohesive energy density as a numerical value indicating the solvency behavior of a specific solvent (equation 2.2)[7].

$$\alpha = \sqrt{c}\left[\frac{A\square - PT}{Vm}\right] {}^{1}\!/_{2} \ldots (2.2)$$

Solubility parameter (α) is a solvent property which measures the work necessary to separate the solvent molecules to create a suitably sized cavity, large enough to accommodate the solute. As a rule, it has been found that a good solvent for a certain non-electrolyte has a 5 -values close to that of the solute.

Since, jojoba oil is a non-polar ester compound which has very long straight chain structure, therefore, any solvent, that has a non-polar compound structure and relatively low solubility parameter will leach more jojoba oil. Solvents such as hexane, diethyl ether, were found to be suitable. Table (2.1) lists several solvents in order of increasing Hildebrand parameter.

Table (2.1): Value of solubility parameter for several solvents [7]

Solvent	3/MPa$^{1/2}$ (SI)
n-Pentane	14.4
n-Hexane	14.9
n-Heptane	15.3
Diethyl ether	15.4
1,1,1 Trichloroethane	15.8
White spirit	16.1
Cyclohexane	16.8
Carbon tetrachloride	18.0
Toluene	18.3
Benzene	18.7
Chloroform	18.7
Acetone	19.7
n-Propyl alcohol	24.9
Ethyl alcohol	26.2
n-Butyl alcohol	28.7
Methyl alcohol	29.7
Water	48.0

3. EXPERIMENTAL APPARATUS AND PROCEDURE

3.1 Preparation of Jojoba Seeds

The jojoba seeds used in this work were come from the farm of Jordan University of Science and Technology (JUST).

The seeds were peeled, crushed, and screened. Different particles sizes are obtained by means of roll mill. The average particle size was calculated using screen analysis procedure, and found to be 1.85 mm. To obtain a smaller particle size we used a home seed grinding machine, therefore, a uniformly particle size of 0.45 mm was resulted from this machine.

Three organic solvents; hexane, diethyl ether, and petroleum ether were used to extract the oil from the seeds. These solvents were selected because of its non-polarity as well as jojoba oil is, low solubility parameter, and are recommended from the previous work.

3.2 Description of the Study Unit and List of the Components

3.2.1 Description

The extraction is the process by means of which it is possible, through dissolution in an opportune extractant, to separate a solid or liquid component from a mixture of solid substances (liquid-solid extraction).

The liquid-solid extraction may he take place through a physical process, or it may include also some chemical reactions.

Examples are the extraction of oil from seeds and from oleiferous fruits (soy beans, colza, peanuts, etc.), of fats from animals, of sugar from beets.

The extraction plant produced by Didacta Italia stands besides the aforementioned extra plants, allowing the conduction in the laboratory of the significant experiences oh the solid-liquid and liquid-liquid extraction methods, either with light and heavy extractants [8].

The plant conceived in this way is composed of: a- An electronically controlled heating unit.

b- A balloon for the extractor liquid winded up by a heater with electric resistances; a column for the vapors, flashed under vacuum c- A condenser on the head with the possibility of effectuating tests under vacuum conditions.

d- A container where it is possible to effectuate the separation, with three kits to be assembled (Dl, D2, D3).

It is then possible to control the temperature of the extractor liquid. For a better didactic utilization, all these components are built in borosilicate glass, so to follow directly all the phases of the extraction process. The whole unit is supported by a sturdy tubular frame, of the assembleable type.

Another advantage of the plant is the extreme versatility on the substitution of the extraction kit; such operation is made easier through one moveable carriage, on which is mounted the container of the kit.

3.2.2 List of Components of the Unit

3.2.2.1 Frame

In galvanized steel, complete with:

- Carriage for the support and the movement of the extraction tank (18).

- Surface for the support of the electrical feeders.
- Support plane for the electric healer.

3.2.2.2 Extraction Column, Entirely Built in Borosilicate Glass (drawing P 1126- B) or figure (3.1)

Ref 1. Balloon for the heating of the extractant and for the gathering of the solute. Ref 2. Connecting column, flashed under vacuum.
Ref 3, Refrigerating head.
Ref 4. Extraction tank head.
Ref 5. Tank with filtering bottom which inserted in the lank "18" allows for solid- liquid extraction.
Ref 6.. Trap for the extraction of the solution in the solid-liquid mode, complete with tap for sample drawing.
Ref 7. Connection on the bottom of the extraction tank with the drawing valve of the heavy phase.
Ref 8. Electric heater.
Ref 9. Feeder 220 V 1000 W for the heating resistance, with manual adjustment of the power.
Ref 10. Feeder 220 V 1000 W for the heating of the heating power.
Ref 11. Supply tank.
Ref 12. Connecting room head of balloon "1” to column ”2".
Ref 13. Solvent recovery valve.
Ref 14. Supply tank valve.
Ref 15. Teflon connecting section arranged to receive the thermoresistance PT100.
Ref 16. Upper connection bridge.
Ref 17. Plug on the head of the column with two rubberholders; it can be utilized to create vacuum conditions within the column.
Ref 18. Extraction tank.
Ref 19. Locking flange to the carriage and to the lid.
Ref 20. Teflon connecting section.
Ref 21. Balloon exhaust valve.
Ref 23. Adapter section for the contact thermometer.
Ref 22-24. Series of sliding connections for the quick adaptation to the various process solutions.
Ref 25. Thermometer with adaptable electric contact.
Ref 27. Shaped vessel which, inserted within the tank "18", allows the execution of the liquid-liquid extraction with light extractant (Solution D3).
Ref 28. 90° pipe fitting for upper bridge.
Ref 29. Shaped vessel which, inserted within the lank "18", allows the execution of the liquid-liquid extraction with heavy extractant (Solution D2).
Ref 30. Bottom connection of the extraction tank with light phase drawing valve.
Ref 31. Glass tube for connection and drain.
Ref 32. - Teflon connecting section arranged to receive the thermo-resistance PT100. Ref 33. Connection cable between the contact thermometer "25" and the feeder "9". Ref 34. Adjustment valve for the cooling water.
Ref 35. Cooling water inlet.
Ref 36. Piping in silicone rubber for cooling circuit.

Note: The real picture and detailed overview of the plant are placed on appendix (B)

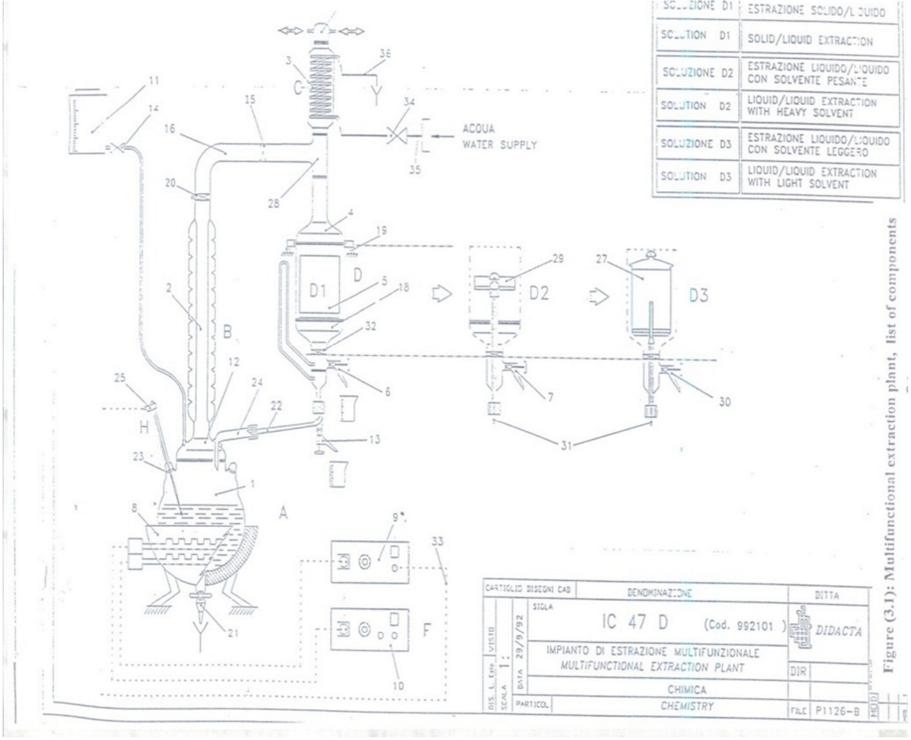

Figure (3.1): Multifunctional extraction plant, list of components

3.3 Operation of the Electrical Feeders "9" and "10"

- At the start of the process, when it is wished to bring to temperature the unit quickly, it is possible to adjust the potentiometer of both the feeders at the maximum value.
- When the temperature reaches the set point prefixed on the thermometer "25", it is possible to decrease the power delivered by both feeders up to when a level is reached which allows the maintain of a constant temperature within the column.
-

3.4 Starting the Unit

1) The extracting liquid has to be put within the balloon " 1", so to fill about 3/4 of its capacity, while in the container "18" has to be positioned the opportunely adapted extraction kit. (Note: the balloon capacity is 10 L).

2) Presetting the heating temperature of the extracting liquid by acting on the control "25" which controls the feeder "9"; this variable power thyristor feeder allows the modulation of the temperature, keeping it constant.

3) Positioning on an average value the other feeder "10", which is a thyristor feeder itself, but with manual control.

4) Checking that the water arrive to the condenser "3" by operating on the valve "34".

3.5 Liquid-solid Extraction

C = Extracting liquid

A+B = Solid mixture from which A is extracted

a) After having assembled the apposite extractor as shown in the drawing P 1126-B, proceed by sending current to the feeders for the heating of the oil bath.

b) The vapors that have been produced from the boiling of the extracting liquid C pass through the column *"2"* and to the cooler "3" where they are opportunely condensed, falling back as fine drops within the container "18" where is positioned the filtering tank (5) which contains the solid mixture A +B to be separated, The liquid C passes through this mixture, thus extracting the component A.

c) This "Operation has to be performed up to when the level of the liquid A + C reaches the top of the, container "18" and overflows into the balloon "1" through the siphon "6".

d) At this point, C+A enter the balloon "1" where C is again distilled, repeating in this way the cycle up to when A is exhausted, gathering all in the balloon "1" [9].

Figure (3.2) shows the process which carried out after starting the unit [9].

The second step is simple distillation to separate the oil from the solvent; the solvent was reused in the next extraction process. The volume of extracted oil was measured, and the oil yield is calculated as follow:

$$\text{Oil yield} = \frac{\textit{Total oil recovered}}{\textit{Total mass of seeds}} \times 100\% \quad \ldots \quad (3\ 1)$$

Figure (3.3) shows the simple distillation apparatus used in this work. Water bath was used; because direct heating destroys the chemical constituents within the jojoba oil.

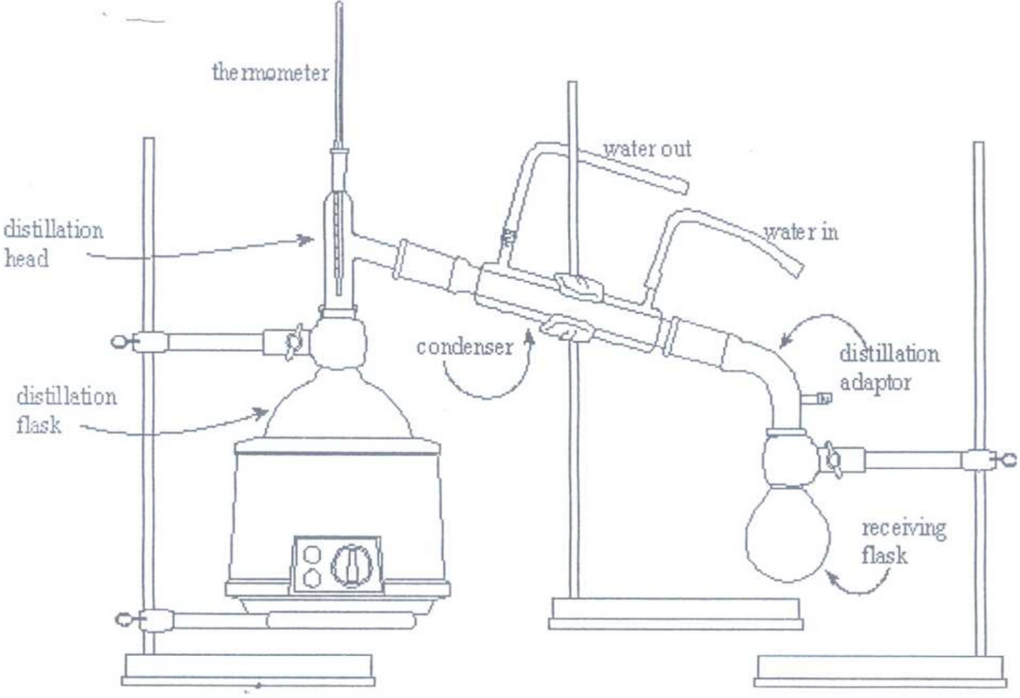

Figure (3.3): Simple distillation apparatus.

Summery of Experimental Procedure

1. Charging the solvent balloon of 7.5 liter of solvent for each run.
2. Filling the solid phase container of 250 g of crushed jojoba seeds.
3. Adjustment the contact thermometer at the set-point which is the boiling point for the solvent.
4. Opening the cooling water valve to be used for condensing the solvent vapor.
5. Operating the two electrical feeders at the maximum value.
6. When the temperature of solvent reaches the set-point (boiling point) prefixed on the thermometer "25", it is possible to decrease the power delivered by both feeders up to when a level is reached which allows the maintain of a constant temperature within the column.
7. This operation has to be performed up to when the level of the liquid in seeds container reaches the top, and overflows into the solvent balloon through the siphon "6".
8. At this point, (solvent + oil) enter the solvent balloon where the solvent is again distilled, repeating in this way the cycle up to when oil is finished, and gathering all in the balloon.
9. Switching off the electrical feeders.
10. The temperature of apparatus gradually decreases and the apparatus get cooled.
11. Emptying the balloon from liquid mixture, and removing the spent solid from the seeds container.
12. Separation of oil from solvent by means of simple distillation.

4. Results and Discussion

4.1 Experimental Data

The objective of this work is to select the best solvent which recovers a highest amount of the oil, and studying the effect of seed particle size on the leaching process.

Table (4.1) compares among the three solvents used in this study.

Solvent	Structure	Boiling point, °C	Specific gravity	Cost of the solvent (JD/L)	Solubility parameter '6 o/MPa'$^{1/2}$
n-Hexane	CeHi4	68.74	0.67	5.60	14.9
Diethyl ether	$(CH_3CH_2)_20$	35	0.71	6.80	15.4
Petroleum ether	Mixture of hydrocarbons	40-60	0.64	6.40	15.4 *

* This value is taken for diethyl ether, since its value isn't available.

For extraction process using n-hexane, three trails of first stage and second stage leaching were operated at a particle size of 1.85 mm, but one run for the particle size of 0.45 mm. For the second leaching process; the spent seeds let to be dried in an ambient temperature for two days, then it was charged to the extraction unit. For diethyl ether and petroleum ether, just one trial was operated; the particle size was 0.45 mm. The following table illustrates the results obtained from n-hexane extraction process which the diameter of seeds was 1.85 mm. From the extracted oil, we measured its density and it was found to be 0.862 g/ml.

Table (4.2): Percentage of jojoba oil after first and second leaching using n- hexane, particle size of 1.85 mm.

Number of trial	Mass of seeds, g	Volume of oil after first stage, ml	Mass of oil after first stage, g	Oil yield after first stage, %	Volume of oil after second stage, ml	Mass of oil after second stage, g	Oil yield after second stage, %	Total oli yield, %
1	250	94	81	32.4	18.5	16	8	40.4
2	250	92	79.3	31.7	21	18	7.2	38.9
3	250	87	75	30.0	20	17.2	6.9	36.9

The next table gives the percentage oil recovered using n-hexane, diethyl ether and petroleum ether. The particle size was 0.45 mm

Table (4.3): Percentage of jojoba oil after leaching using n-hexane, diethyl ether, and petroleum ether, particle size of 0.45 mm.

Solvent	Mass of seeds, g	Volume of oil, ml	Mass of oil, g	Oil yield, %
n- Hexane	250	155	133.6	Highest one
Diethyl ether	250	150	129.3	The second
Petroleum ether	250	114	98.3	The third

In figure (4.1), we show the amount of oil recovered using the three solvents, with respect to particle diameter of 0.45mm.

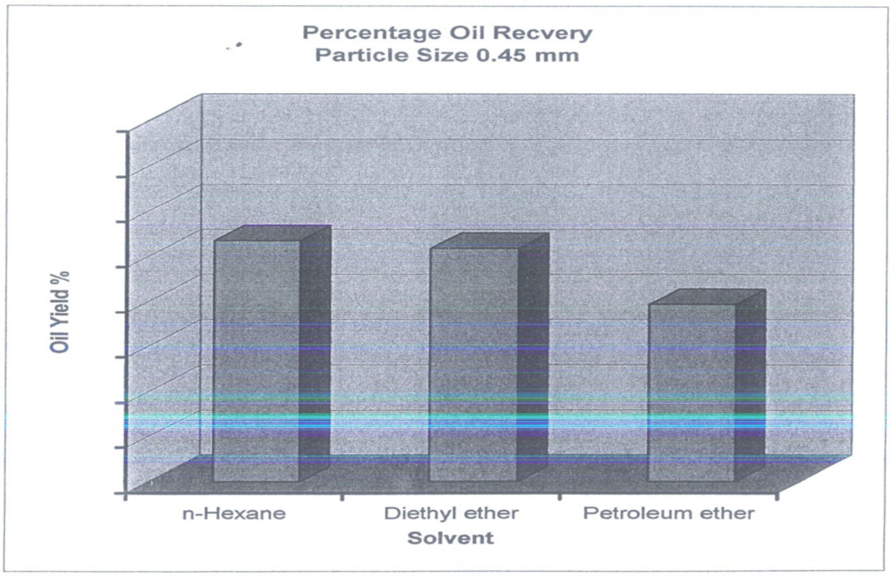

Figure (4.1): Percentage oil recovery, particle size of 0.45 mm.

A comparison of operating conditions in the process and two physical properties of leached oil among the three solvents will give us meaningful information about the best solvent. The operating conditions studied are: cooling water requirement, time required for leaching, and solvent losses. The two physical properties are odor of solvent in leached oil, and color of leached oil. All these factors are given in table (4.4).

Table (4.4): Comparison among the three solvents.

Solvent	Cooling water requirement, L/min	Time required for leaching*, min	Solvent losses for each run	Odor of solvent in leached oil	Color of leached oil
n-Hexane	3 (enough)	55	Less than 4 %	Needs purification to remove	Clear yellow liquid
Diethyl ether	15 (not enough, needs more)	30	About 40 %	No odor after 5 days	Yellow liquid with some suspended matter (turbidity)
Petroleum ether	15 (approximately enough)	40	Less than 4 %	No odor after 5 days	Yellow liquid with some suspended matter (turbidity), but less than diethyl ether

* Time required was recorded from which the first condensed solvent vapor drop was formed until the oil was exhausted.

4.2 Selection of the Best Solvent

From the tables (4.1, 4.2, 4.3, and 4.4); we can conclude that the **n-hexane** is the most suitable solvent for using in jojoba oil extraction, its advantages over other solvents are:

1- Has the highest oil yield.
2- Has the lowest cost.
3- Operated with small amount of cooling water.
4- Maas losses of the solvent are the minimum during the extraction and distillation processes.
5- Produces a clear yellow liquid.

On the other hand, n-hexane has one disadvantage; it is the odor which remains in the oil, the proposed processes for removing the n-hexane odor from the oil after simple distillation are:

1. Vacuum distillation.
2. Rotary evaporator method.
3. Stripping steam.

4.3 Effect of Particle Size of Jojoba Seeds

From these results, it can be seen that the reduction in particle diameter has a positive influence on the leaching process. Hence; when the particle diameter decreases, the jojoba oil yield increases.

When the particle diameter was reduced from 1.85 to 0.45 mm using n-hexane as a solvent, the variation in oil yield was significant (increase of oil yield about 22 % after first stage and about 15 % after first and second stage leaching). This variation ^ comes from the decreasing in internal mass resistances of the seeds.

The conclusion can be drawn that when the particle size is very small, the leaching process will be enhanced and lead to rapid oil yield and finally reach the maximum value. This is due to the reduction in resistances which has an effect on diffusion of the solvent inside the particles and diffusion of solute from the particles to the surface of the particles. This also could be due to breaking of the cellular structure of particles. Reduction in particle size will contribute to the breakage of the jojoba seed's cellular structure. This results in reduction in the internal resistance, responsible for the solute which penetrates the cells and leaches the oil out. As a result, this breakage will increase the oil yield rate and overall oil yield.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Experimental data are showed that the batch leaching process of jojoba oil using multifunctional extraction plant from Didactics Italia Company is very sensitive to operating parameters, such as type of solvent, and particle size of jojoba seed.

From the results, it can be concluded that the solubility of oil in a solvent depends strongly on the polarity of both oil and solvent. The more non-polarity of the solvent leads to larger amount of oil yield, and the less value of solubility parameter gets the same result. n-Hexane is the most appropriate solvent used in this work since it produces the higher oil yield than that yield of diethyl ether and petroleum ether, and because of the most important factors which are the relative low cost per liter and it doesn't need high rat£ of cooling water.

The second conclusion is that for particle size, it can be seen that the reduction in particle diameter has a positive influence on the leaching process. Hence; when the particle diameter decreases, the jojoba oil yield increases.

It can be also concluded that the Jordanian jojoba seeds contains about 50 % recoverable oil.

Recommendations

1. For the modification of the multifunctional extraction plant:
   a) A stirrer may be inserted into the seeds' container to speed up the leaching rate, extracting more oil, and decreasing the total time of the process.
   b) Operate the unit under vacuum conditions by employing the vacuum accessories at the top of the condenser.
   c) The unit must be directly connected with distillation unit to avoid solvent losses.

parameter, thus minimizing heat and cooling water requirements and maximizing oil yield.

3. For removing the n-hexane odor from the oil after simple distillation, we are proposed the following methods:
   a) Vacuum distillation.
   b) Rotary evaporator method.
   c) Stripping steam.
   d) Adsorption or activated carbon.

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