FACTORS AFFECTING THE EXTRACTION PROCESS

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ABSTRACT

This work was conducted to study the effect of factors influencing the extraction process of oil-bearing flakes of sunflower, cotton, and soybean seeds by using the commercial hexane.

These factors were the moisture content, flake thickness, extraction time, temperature of extraction, solvent to seed ratios, and foreign matter for sunflower, cotton, and soybean seeds. The lint and the smut were studied also for the cotton seeds only.

It was found that the moisture content of 9-11%, temperature of extraction of 60-65 °C, flake thickness of 2-3 mm, five hours extraction time, and 1 to 1 seed to solvent weight ratio could be recommended as the best conditions for the extraction process. Cotton seed lint and smut should be low as much as possible.

INTRODUCTION

The solvent extraction process in its simplest term means the recovery of oil from oil-bearing materials by making use of a suitable type of volatile solvent (1). It is a mass transfer operation in which materials are transported from one phase to another for the purpose of separating one or more compounds from mixture. In the case of oil seeds extraction, crude vegetable oil is dissolved in a solvent to separate it from the insoluble meal that is primary composed of protein and carbohydrates.

Oil is produced from oil seeds by mechanical pressing or extraction with solvent, or combination of both. The residues after extraction called vegetable meals were at one time unwanted by-product. Meals are now recognized as important source of protein, and are used in large quantities as animal foodstuffs. Many types of oil seed exist in nature and are extracted with solvent. The amount of oil in the seed varies from 15-55 wt.% (2).

Various methods of extraction employed have their own degrees of limitations, but all of them have common objectives such as to obtain the oil in its native form, the final yield of the oil should be maximum, and to produce the residual oil cakes in such a form that the proteins and other non-lipids have the greatest possible value (3).

Various liquids have been used as solvents for extraction by the oil seeds industry. Mixtures of hexanes, rich in n-hexane, have been the solvents of choice by the oil seed processing industry for the past 50 years. Listing of references for various solvents and their usage were published on two occasions (4). Hron (5) reviewed bio renewable solvents, and Johnson and Lusas (6) compared properties of alternative solvents. Over 70 solvents have been mentioned in scientific literature as being used in oil seed extraction (7).

The work deals with studying the factors affecting the extraction process of sunflower, cotton, and soybean flakes extracted by commercial hexane.

EXPERIMENTAL WORK

Materials

Sunflower, cotton, and soybean Seeds

Composition of sunflower, cotton, and soybean Seed are shown in Table (1) (8, 9).

Commercial Hexane

Commercial hexane [produced by The State Company for Gas Manufacture (AL-Taji)] is used as a solvent for oil-seed extraction. The composition of the commercial hexane is shown in Table (2).

Table (1) Composition of Sunflower, Cotton, and Soybeans.

Properties	Type of Seed		
	Sunflower	Cotton	Soybean
Oil Content, (%)	42	20	19
Moisture, (%)	3.8	6.6	6.8
Foreign Matter, (%)	3.1	2	1.2
Protein, (%)	32.64	38	45.62
Solidification point, (°C)	- 15	0	- 8
Iodine value	110-145	99-117	120-143
Water, (%)	4	7	13
Fibers, (%)	3	3	6
Ash, (%)	1	5	5
N-free extract, (%)	12	15	10
Smut, (%)	---	10.6	---
Lint, (%)	---	13.4	---

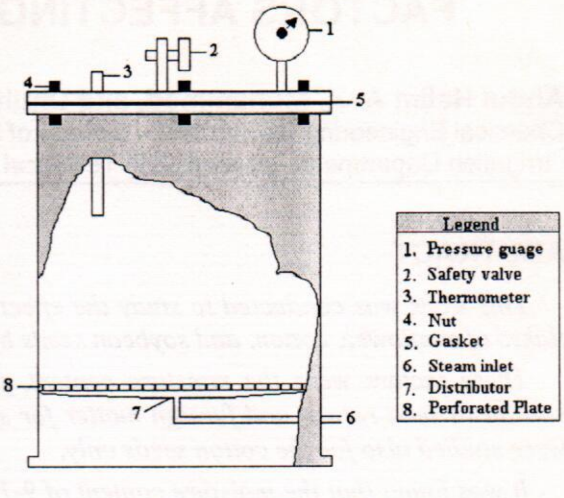

Figure (1) The conditioner

Table (2) Composition of Commercial.

Component	Commercial hexane
Pentane (C_5)	1
2-2-Di-methyl Butane	0.2
2-Methyl Pentane	23.2
3-Methyl Pentane	18.5
N. hexane	46.9
Methyl cyclopentane	7
Benzene	1.5
Cyclobenzene	1.7
Heptane (C_7)	---

Experimental Procedure

Sieving

A sieve of 2mm was used for screening the seeds.

The Conditioner

The conditioner (Figure 1) used for cooking the seed. This conditioner was an autoclave of stainless steel 120 mm diameter, and 220 mm height. A distributor (50 hole, 1mm hole diameter) for steam injection was placed 50 mm at the bottom of the cylinder over which a perforated plate was fixed to rest seeds on. The cover for the cylinder is a stainless steel cap of 150mm diameter, and 20 mm height, containing a pressure gauge of 5×105 N/m2, a safety valve, and a thermometer of 150°C. The cap and the cylinder were separated by a gasket and tighten strongly to each other with five nuts to prevent leaks.

The Flaking Machine

The flaking machine (Figure 2) consists of two smooth rubber rolls 50 mm diameter, and 380 mm long adjusted at variable distance, moving in opposing direction driven by hands. A glass container at the bottom receives the flaked seeds.

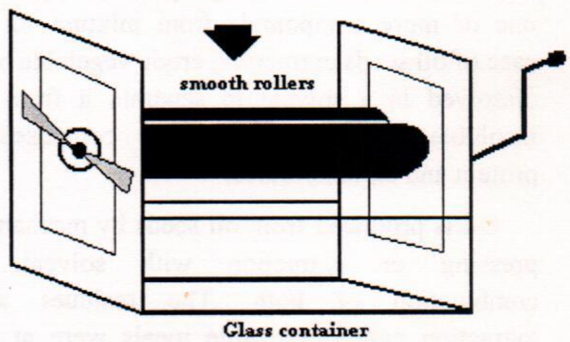

Figure (2) The Flaking Machine.

The Soxhlet Extractor

The soxhlet extractor (Figure 3) used for extracting flaked seeds to produce oil and meal. It consists of a heating mantel 1.2 KW, 250 ml with two necked round bottom flask, a thermometer of 250°C in one neck and a glass soxhlet (3.5 mm diameter and 26 mm height) fitted to the other, inside which a thumble supporting the flaked seeds is fixed. A reflecting cooling water glass condenser (3.5 mm diameter and 21 mm height) fitted to the soxhlet for solvent vapour condensation.

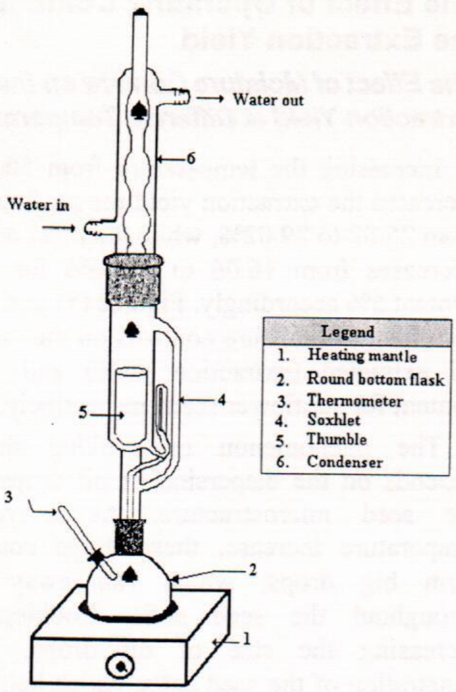

Figure (3) The Soxhlet Extractor

The Vacuum Distillation Unit

A typical assembly due to Wendland (10) is depicted in Figure (4). It consists of a heating mantel 1.2 KW, 250 ml with two necks round bottom flask, a thermometer of 250°C fitted in one neck and a glass column 50 mm diameter, 300 mm long fitted to the other neck. A glass condenser (3.5 mm diameter and 500 mm long) is connected to the distillation column using water as a cooling media. The whole system is evacuated by means of a vacuum pump connected between the condenser and the receiver.

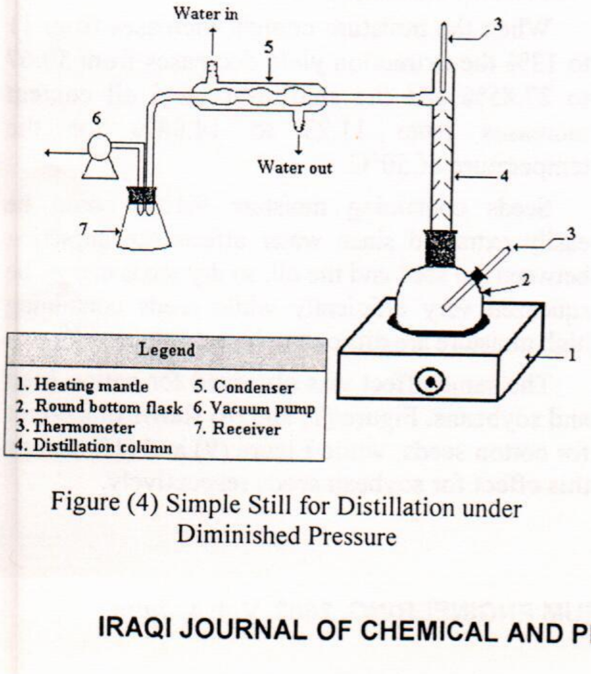

Figure (4) Simple Still for Distillation under Diminished Pressure

Operating Conditions

The Effect of Moisture Content

Samples (10 gm) of sunflower, cotton, and soybean seeds were prepared by conditioning them at temperature of 90°C and pressure of 2×105 N/m2 for different moisture contents (5,7,9,11,12, and 13%), and then the samples were flaked. The flaked seeds were extracted in a soxhlet for five hours and at different temperatures (50,60, and 70°C) for each type of seeds and for each of moisture content.

The Effect of Flake Thickness

Samples (10 gm) of sunflower, cotton, and soybean seeds were prepared by the cooking method for moisture content of 9%. The cooked seeds were flaked to thickness of 5, 3, 2, 1, and 0.5 mm. The flaked seeds were extracted in a soxhlet for five hours at 65°C.

The Effect of Extraction Time and Seed to Solvent Ratio

Samples (10 gm) of sunflower, cotton, and soybean seeds were prepared by the cooking method at 9% moisture content, flake thickness of 2-3 mm and temperature of 65°C. The flaked seeds were extracted in a soxhlet for extraction time of 60, 120, 240, and 360 min. and seed to solvent ratios of 1:1, 1:5, and 1:10 on wt basis for five hours.

The Effect of Solvent Temperature

Samples (10 gm) of sunflower, cotton, and soybean seeds were prepared by the cooking method at 9% moisture content, flaking them to 2-3 mm, and extracting them in a soxhlet at 40, 50, 60, and 70°C for five hours extraction time.

The Effect of Foreign Matter

Samples (10 gm) of sunflower, cotton, and soybean seeds of different percentages of foreign matter {0 (meat only), 2, 5, and 10%} were prepared by the cooking method at 9% moisture content, and flaking them to 2-3 mm. The flaked seeds were extracted in a soxhlet for five hours at 65°C.

The Effect of Cotton Seeds Lint

Samples (5 gm) of cotton seeds were immersed in sulfuric acid 98% for 5, 10, 20, and 30 sec., then washed directly with excessive amount of water. The washed seeds dried in a 110°C oven for two hours. The dried seeds were conditioned, flaked, and extracted.

The Effect of Cotton Seeds Smut

Samples (10 gm) of cotton seeds were broken using a cutter. The smutted seeds were collected, weighed, conditioned, and flaked then extracted.

RESULTS AND DISCUSSION

Chemical Analysis of Sunflower, Cotton, and Soybean Seeds

Tests that were carried out for the raw seeds used in this investigation (A.O.C.S methods) showed their chemical analysis in Table (3).

The allowable limit for moisture content is 6-10% for sunflower seeds, 8.5-10% for cotton seeds, and 7% for soybean seeds. So it is clear that the moisture content for the three types of the seeds is below the allowable limits.

The allowable limit for foreign matter is 5-15% for sunflower seeds, 0-4% for cotton seeds, and 0.5-2% for soybean seeds. So it is clear that the foreign matter for the three types of the seeds is within the allowable limits. (Ref. operating manual of the General Company for Vegetable Oil Industry).

Chemical Analysis of Commercial Hexane

The chemical analyses of commercial is shown in Table (3).

Table (3) Chemical analysis of commercial hexane

Property	Commercial hexane	ASTM designation of test method
Sp. Gravity @ 60/60°F	0.674	D- 1298
Color	25+	D- 156
Doctor Test	Pass	D- 484
Vapour Pressure, max, psia. at 100°F	0.43	D- 323
Distillation		D- 1078
Aromatics (C_6), wt%	2.23	GC
I.B.P., °C	62	
50% vol. Recovery, °C	at 64	
90% vol. Recovery, °C	at 69	
Dry point, °C	70	

The Effect of Operating Conditions on the Extraction Yield

The Effect of Moisture Content on the Extraction Yield at Different Temperatures

Increasing the temperature from 50 to 70°C increases the extraction yield for sunflower seeds from 25.82 to 29.02%, while the meal oil content decreases from 16.06 to 12.96% for moisture content 5% accordingly. Figures (5) and (6) show the effect of moisture contents on the quantity of oil extracted (extraction yield) and meal oil content for sunflower seeds respectively.

The phenomenon of cooking the seeds depends on the dispersion of oil drops through the seed microstructure. As a result of temperature increase, these drops coalesce to form big drops, which run away quickly throughout the seed cells. Cooking causes increasing the size of oil drops, and the penetration of the seed cells, which helps the oil to flow. Cooking also cause a reduction in the oil attraction to the solid surfaces of the seed to get a good extraction yield when using pressure.

This is true for all other temperatures at different moisture contents. When the moisture content increases from 5 to 9%, the extraction yield increases from 25.82 to 31.32% and the sunflower meal oil content decreases from 16.06 to 10.55% for the temperature of 50°C. Since heating plays an important role, so it must be taken in care.

When the moisture content increases from 9 to 11% the extraction yield decrease from 31.32 to 30.67%, while meal oil content increase from 10.55 to 11.23% since water (as a polar substance) hinders wetting of the seed surface and penetration of solvent into the seed. It reduces the diffusion coefficient [11].

When the moisture content increases from 11 to 13% the extraction yield decreases from 30.67 to 27.85% and the sunflower meal oil content increases from 11.23 to 14.08% for the temperature of 50°C.

Seeds containing moisture 9-10% could be easily extracted since water affects the attraction between the seed and the oil, so dry seeds cannot be squeezed very efficiently while seeds containing high moisture are difficult to be extracted [1, 7, 12].

The same effect was observed for cotton seed and soybeans. Figure (7) and (8) shows this effect for cotton seeds, while Figure (9) and (10) shows this effect for soybean seeds respectively.

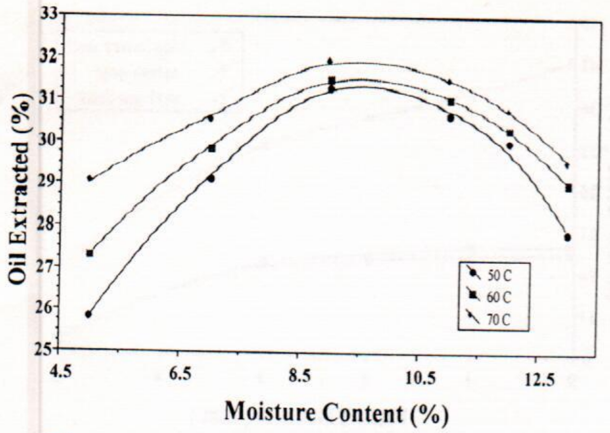

Figure (5) the relationship between the moisture content and the oil extracted for sunflower seeds at different temperatures.

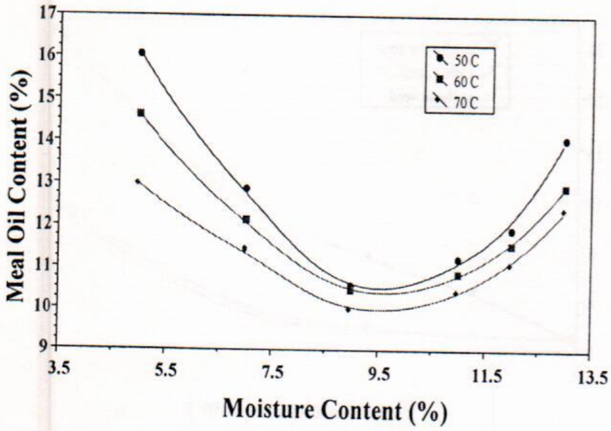

Figure (6) the relationship between the moisture content and the meal oil content for sunflower seeds at different temperatures

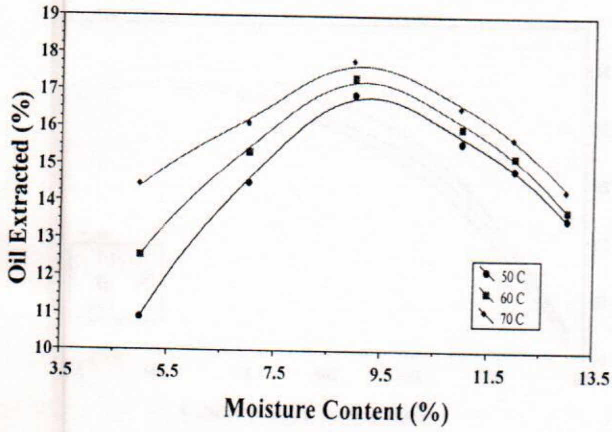

Figure (7) the relationship between the moisture content and the oil extracted for cotton seeds at different temperatures

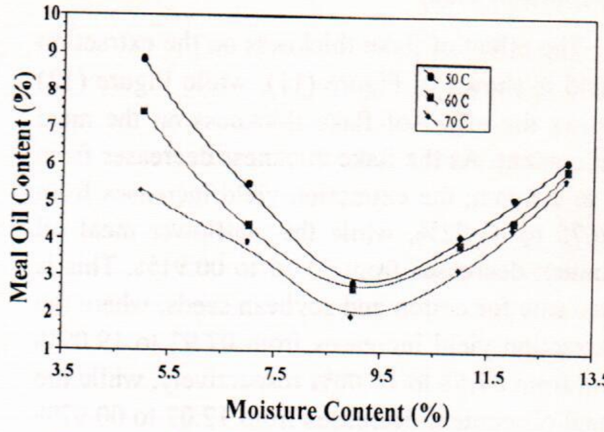

Figure (8) the relationship between the moisture content and the meal oil content for cotton seeds at different temperatures

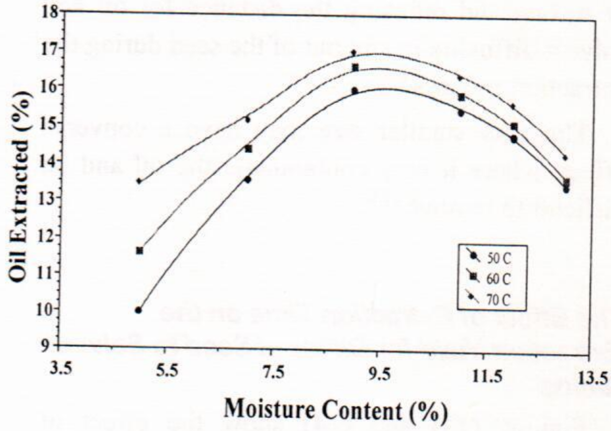

Figure (9) the relationship between the moisture content and the oil extracted for cotton seeds at different temperatures

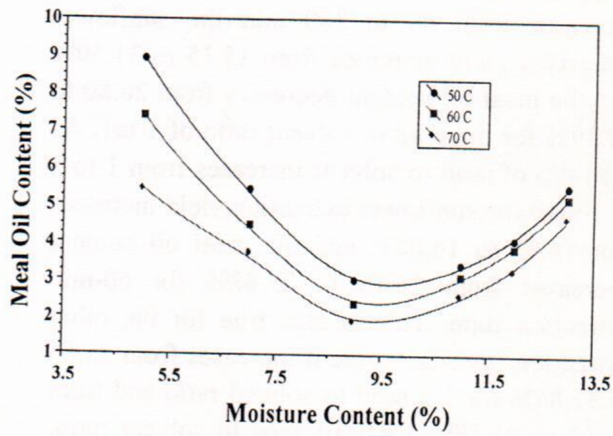

Figure (10) the relationship between the moisture content and the meal oil content for soybean seeds at different temperatures

The Effect of Flake Thickness on the Extraction Yield

The effect of flake thickness on the extraction yield is shown in Figure (11), while Figure (12) shows the effect of flake thickness on the meal oil content. As the flake thickness decreases from 5 to 0.5 mm, the extraction yield increases from 16.75 to 41.02%, while the sunflower meal oil content decreases from 25.15 to 00.91%. This is also true for cotton and soybean seeds, where the extraction yield increases from 07.97 to 19.09% and from 07.58 to 18.00% respectively, while the meal oil content decreases from 12.02 to 00.97% and from 11.40 to 00.98% for cotton and soybean seeds respectively.

Rolling seeds into thin flakes facilitates solvent extraction because of the disruptive effect of rolling and reducing the distance for oil and solvent diffusion in and out of the seed during the extraction process [7].

The very smaller size may have a converse effect, where it may contaminate the oil and be difficult to remove [13].

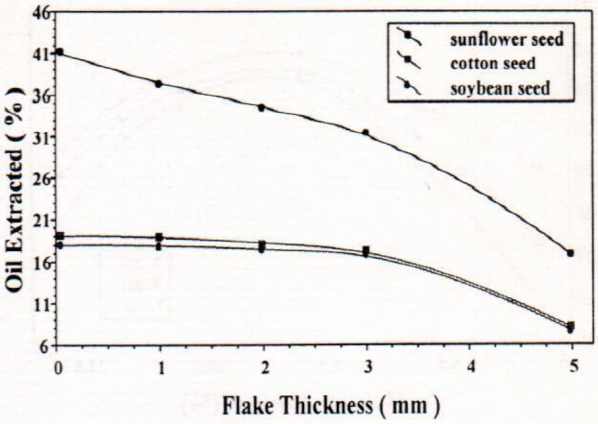

Figure (11) the relationship between the flake thickness and the oil extracted for sunflower, cotton, and soybean seeds

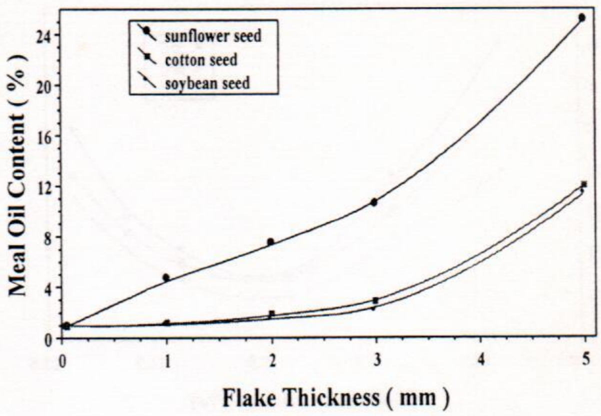

Figure (12) the relationship between the flake thickness and the meal oil content for sunflower, cotton, and soybean seeds

The Effect of Extraction Time on the Extraction Yield for Different Seed to Solvent Ratios

Figures (13) and (14) show the effect of extraction time on the extraction yield and meal oil content for different seed: solvent ratios for sunflower seeds.

It is clear that when the extraction time increases from 60 to 360 min the sunflower extraction yield increases from 15.15 to 31.50% and the meal oil content decreases from 26.80 to 10.39% for the seed to solvent ratio of 1 to1. As the ratio of seed to solvent increases from 1 to 5 to 1 to10 the sunflower extraction yield increases from15.85 to 16.02% and the meal oil content decreases from 26.13 to 25.88% for 60-min extraction time. This is also true for the other extraction periods, where it increases from 15.85 to 31.84% for 1:5 seed to solvent ratio and from 16.02 to 32.18% for 1:10 seed to solvent ratio. While sunflower meal oil content decreases from 26.13 to10.06% for 1:5 seed to solvent ratio and from 25.88 to 09.81% for 1:10 seed to solvent ratio.

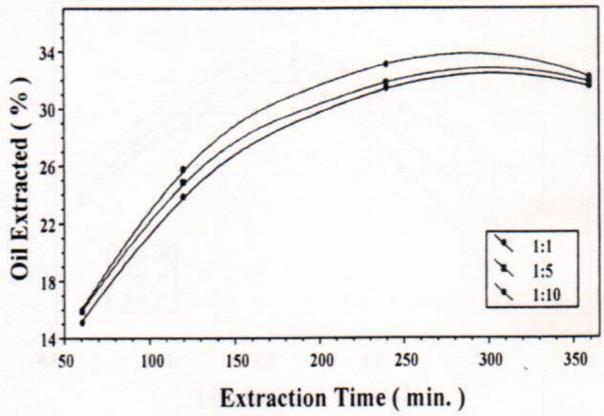

Figure (13) the relationship between the extraction time and the oil extracted for sunflower seeds at different seed : solvent ratios

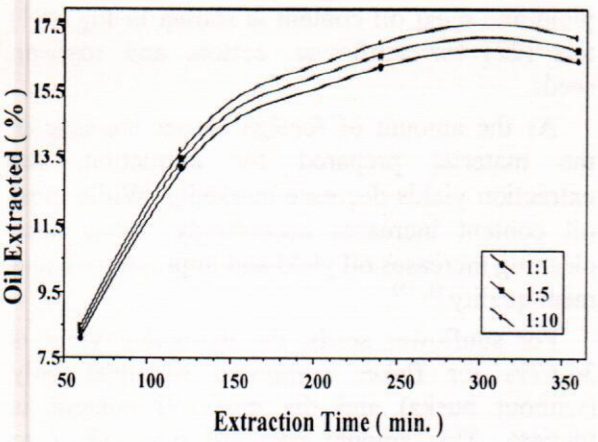

Figure (14) the relationship between the extraction time and the oil extracted for cotton seeds at different seed : solvent ratios

This also fits the cotton and soybean seeds. Figures (15) and (16) show this effect for cotton seeds, while Figures (17) and (18) show this effect for soybean seeds.

When the temperature and extraction time were kept constant, the quantity of oil extracted depends on the amount of solvent to certain extent. To get a residual oil less than 1%, it is necessary to reach the highest time required.

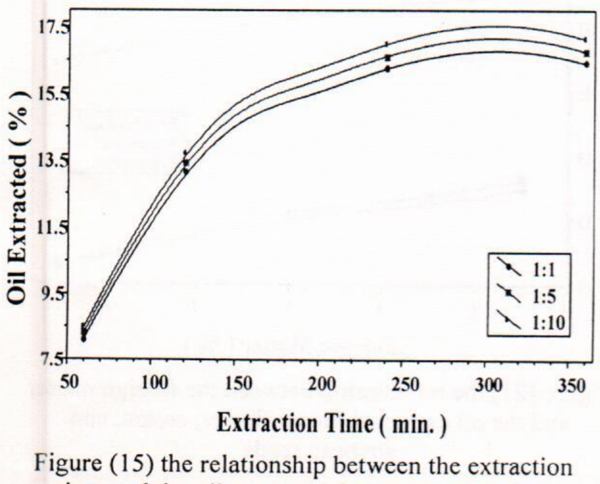

Figure (15) the relationship between the extraction time and the oil extracted for soybean seeds at different seed : solvent ratios

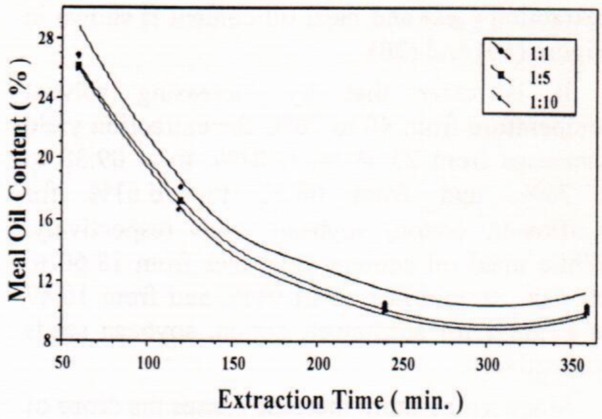

Figure (16) the relationship between the extraction time and the meal oil content for sunflower seeds at different seed: solvent ratios

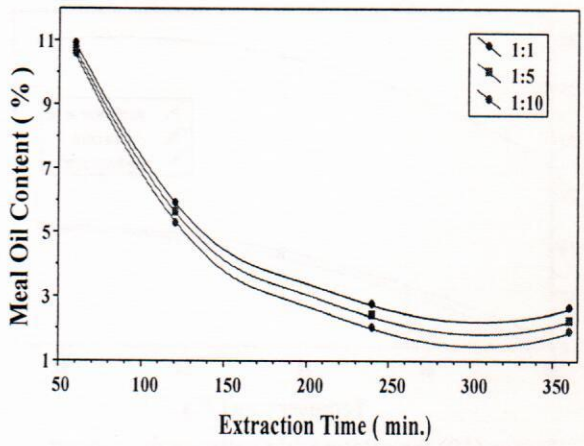

Figure (17) the relationship between the extraction time and the meal oil content for cotton seeds at different seed: solvent ratios

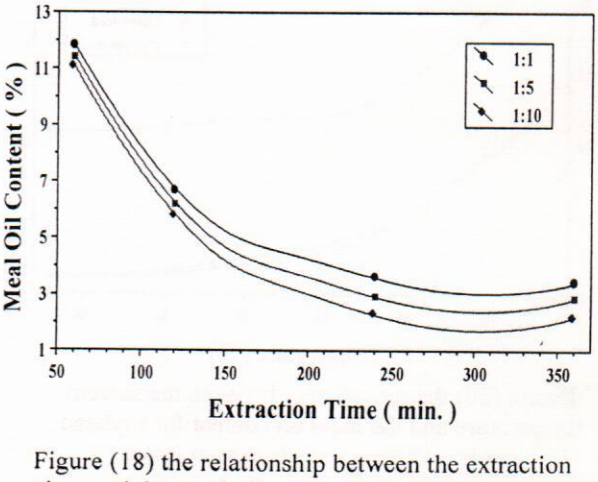

Figure (18) the relationship between the extraction time and the meal oil content for soybean seeds at different seed: solvent ratios

The Effect of Extraction Temperature on the Extraction Yield

The effect of extraction temperature on the extraction yield and meal oil content is shown in Figure (19) and (20).

It is clear that by increasing solvent temperature from 40 to 70°C the extraction yield increases from 23.38 to 32.07%, from 09.38 to 17.76%, and from 08.50 to 16.61% for sunflower, cotton, soybean seeds respectively. While meal oil content decreases from 18.60 to 09.77%, from 10.61 to 01.94%, and from 10.47 to 01.88% for sunflower, cotton, soybean seeds respectively.

Since temperature increase causes the drops of oil to coalesce to form big drops, which runs away quickly through out the seed cells [7, 14]. So oil could be extracted easily from the seeds, by increasing temperature more than 50°C [7].

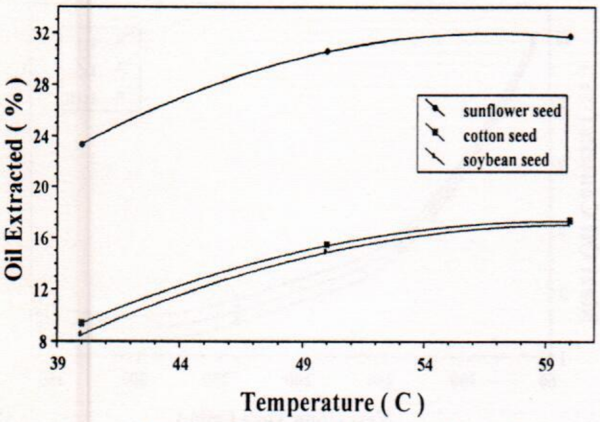

Figure (19) the relationship between the solvent temperature and the oil extracted for sunflower, cotton, and soybean seeds

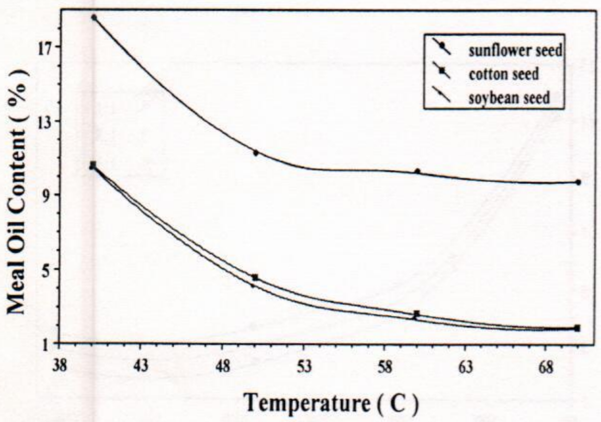

Figure (20) the relationship between the solvent temperature and the meal oil content for soybean seeds at different seed: solvent ratios

The Effect of Foreign Matter on the Extraction Yield

The effect of foreign matter on the extraction yield and meal oil content is shown in Fig. (21) and (22) for sunflower, cotton, and soybean seeds.

As the amount of foreign matter increase in the material prepared for extraction, the extraction yields decrease markedly. While meal oil content increases accordingly. Since seed cleaning increases oil yield and improving oil and meal quality [1, 15].

For sunflower seeds, the extraction yield is 34.67% for flakes composed of meat only (without husks) and the meal oil content is 08.04%. This amount decrease from 33.27 to 27.50% and the meal oil content increase from 08.65 to 14.48% as the foreign matter increase from 2 to 10%.

This is also true for cotton and soybean seeds. The extraction yield for cottonseed is 16.69% for the meat flakes and the meal oil content is 03.31%. This extraction yield decrease from 15.15% to 08.83% and the meal oil content increase from 04.82 to 11.15% as the foreign matter increase from 2 to 10%.

For soybean seeds, the extraction yield is 15.85% for flakes composed of meat only (without husks) and the meal oil content is 03.14%. This amount decrease from 14.39 to 08.38% and the meal oil content increase from 04.60 to 10.60% as the foreign matter increase from 2 to 10%.

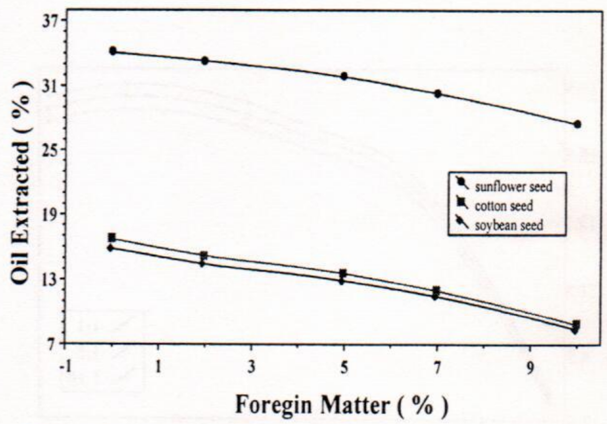

Figure (21) the relationship between the foreign matter and the oil extracted for sunflower, cotton, and soybean seeds

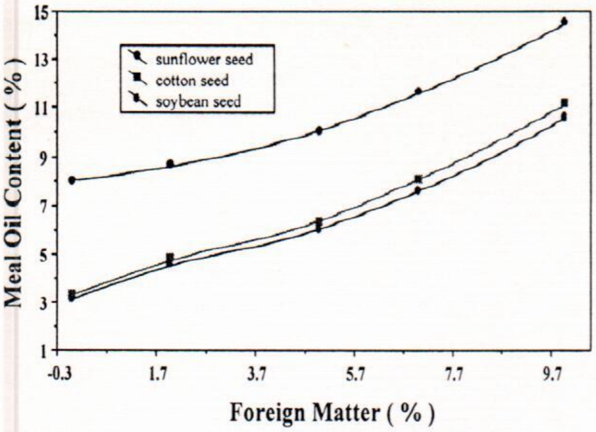

Figure (22) the relationship between the foreign matter and the meal oil content for sunflower and cotton seeds

CONCLUSIONS

From this research it may be concluded that the best conditions for extraction of sunflower, cotton, and soybeans were:

1. Moisture content for conditioning the seeds 9-11%
2. Flake thickness of 2-3 mm.
3. Economic seed: solvent ratio of 1: 1.
4. Temperature of the extraction 60-65C for pure hexane.
5. The extraction time of 5 hr.

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