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Extraction and Characterization of Linden Tree Seed Oil Grown in Malatya with Different Solvents

Yunus ÖNAL^{*1}, Tuğce Begüm MUMCU¹, Tuğba UTKU²

¹ Inonu University, Engineering Faculty, Chemistry Engineering Department, Malatya, Turkey ² Inonu University, Scientific and Technological Research Centre, Malatya, Turkey Yunus ONAL, ORCID No: 0000-0001-6342-6816, Tugce Begüm MUMCU, ORCID No: 0000-0003-1486-2931, Tugba UTKU, ORCID No: 0000-0001-7034-2725 *Corresponding author e-mail: yunus.onal@inonu.edu.tr

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Keywords Fatty acids, Linden seed, Solvent, Soxhlet **Abstract:** In this study, the characterization study of the oil of linden seeds obtained by the extraction method was carried out. The outer shells of linden seeds collected from Malatya region in August 2018 were cleaned before extraction. Hexane, ethyl alcohol and acetone were used as solvents. Elemental analysis, XRD (X-Ray Diffraction), SEM (Scanning Electron Microscopy), ash determination and saponification number determinations were performed for characterization processes. After the analysis, the oil yield with ethyl alcohol was determined as 33% by weight with acetone, 28.65% for hexane and 28% for acetone. In linden seed fatty acid, it was determined as oleic acid: 27.442 for acetone, 30.852 for hexane, 10.955 for acetone by weight, 11.929 for hexane, linoleic acid: 51.188 for acetone, 44.145 for hexane. The soap number value was determined as 232.48 mg KOH/g for ethyl alcohol, 176.72 mgKOH/g for hexane and 246.94 mg KOH/g oil for acetone. While the ash determination value was 5.755% for the seed, the shell ash was determined as 2.1% differently.

Farklı Çözgenlerle Malatya'da Yetişen İhlamur Ağacı Tohumu Yağı Eldesi ve Karakterizasyonu

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Anahtar Kelimeler Çözgen, Ihlamur tohumu, Sokslet, Yağ asitleri **Öz:** Bu çalışmada Ekstraksiyon metodu ile elde edilen Ihlamur tohumlarının yağının karakterizasyon çalışması yapıldı. 2018 yılının Ağustos ayında Malatya yöresinden toplanan ıhlamur tohumlarının dış kabukları ekstraksiyon öncesi temizlendi. Çözücü olarak hekzan, etil alkol ve aseton kullanıldı. Karakterizasyon işlemleri için element analizi, XRD (X-Işınları Difraksiyonu), SEM (Taramalı Elektron Mikroskobu), kül tayini ve sabunlaşma sayısı tayinleri yapıldı. Analiz sonrası etil alkol ile yağ verimi ağırlıkça aseton ile yağ verimi % 33, hekzan için %28.65 ve asoton için yağ verimi % 28 olarak tayin edildi. Ihlamur tohumu yağ asidinde ağırlıkça oleik asit: aseton için 27.442, hekzan için %30.852, ağırlıkça palmitik asit: aseton için %10.955, hekzan için 11.929, ağırlıkça linoleik asit: aseton için 51.188, hekzan için 44.145 olarak tayin edildi. Sabun sayısı değeri ise etil alkol için 232.48 mg KOH/g, hekzan için 176.72 mgKOH/g ve aseton için 246.94 mg KOH/g yağ olarak tayin edildi. Kül tayini değeri tohum için %5.575 iken kabuk külü farklı olarak %2.1 olarak tayin edildi.

1. Introduction

People's eating habits have changed over time. Studies have proven that there is a relationship between their eating habits and the diseases they encounter. Especially in developed countries, people who want to lead a healthy life are more attentive to their nutrition. Carbohydrates, proteins and fats are the main energy sources for the maintenance of human existence. Fats are important essential components for humans and animals. In addition, they give high energy (Gizlenci & Acar, 2019).

People need a total of 2800-3000 calories to carry out their daily activities. It meets 30-35% (850-900 calories) from fats. Considering that 1 g of fat provides 9.3 calories of energy, a person should consume 95 g of fat per day. According to the normal nutrition rules, 1/3 of the fat needs of people can be met as liquid meals, 1/3 as solid fat at breakfast and 1/3 with foods such as cheese, milk and hazelnuts. Fats high in saturated fatty acids pose a great danger to human health. Especially animal-derived oils contain high levels of saturated fatty acids. For this reason, people should obtain at least 30% of the oil they need from vegetable oils (Gizlenci & Acar, 2019).

Vegetable oils formed as a result of obtaining oilseeds in crude oil plants; It is important not only for human nutrition but also for our health. The physical and chemical properties of oils are determined by the ratios and compositions of the fatty acids in them. The composition of oil plants can change constantly. We can list ecological, morphological, physiological and genetic reasons among the reasons for its change. Knowing the factors that cause this is important for the quality of the oil. In addition to these, these factors are also important since the quality of the oil determines the distribution of fatty acids in the oil with its nutritional, technological and processing values. Determining the composition of fatty acids is important in terms of producing oils according to their intended use. Thus, oils suitable for the purpose are produced (Sarwar et al., 2013).

With the increasing population in the world, the amount of oil produced is insufficient to meet the amount of oil required for humans. Turkey's annual vegetable oil production has been researched as 1 million 354 thousand tons. 44% of vegetable oils in our country come from sunflower, 16% from soybean, 13% from cottonseed, 11% from olive, 9% from rapeseed and the remaining 7% from other oil crops such as corn, linseed and sesame is produced. We can list the most important industrially important oilseeds as cotton seed, soybean, sunflower, sesame, peanut, canola, safflower, olive, coconut, palm seed and flaxseed. According to researches; Almost all of the oilseed plants except palm and coconut can be grown in our country in summer or winter. However, our existing oil plants and produced oils cannot meet the consumption. For this reason, over billions of dollars are imported from crude and refined oils, as well as oilseed meal, every year. With the increase in population, Turkey's need for vegetable oil is increasing every year. Therefore, studies on new oil crops should be given importance in order to meet the deficit of this need (Gizlenci & Acar, 2019; Tosun & Özkal, 2000).

In order to obtain oil from oilseeds in industry, it must be subjected to some pre-treatment. To list a few; In hard shells, after core removal, and other oilseeds, including sunflower seeds, direct, cold pressing and/or solvent extraction methods are used. Here, degreased materials such as sunflower seeds are widely used in the animal feed industry. In alternative extraction methods, we can also see aqueous enzymatic extraction and supercritical fluid extraction methods.

It has been experimentally proven that natural oils preserve all their beneficial components compared to other refining oils, that is, they do not spoil their natural structure. Consumers and manufacturers prefer natural oil more than other oils. Apart from olive oil, it has started to take its place in the market in different natural oils. Among these, we can list oils such as rapeseed, sunflower, avocado, hemp, pumpkin seeds and argan. Only these oils are both higher in price and wider in price ranges compared to others. The reason for the high prices of such gourmet oils is that they are obtained by cold pressing method. The low oil yield in cold pressing causes its price to be high. In regions with high humidity, it causes great losses as the fatty acidity increases. It is an important issue to be considered in production. Oils obtained from oilseeds are also used in industry. At the beginning of the widely used sectors; soap, shampoo, fabric dyes, paper, glass paste, cosmetic products, construction materials, medicine, disinfectant, shampoo, plastic industry areas (Salamatullah et al., 2021; Arioğlu et al., 2010; Acaravcı & Ergüven, 2015; Çil et al., 2016). In addition, there are various and important areas of use such as vegetable-derived oils, fatty acid alcohols in the chemical industry, heavy metal soaps, molding agents, lubricant chemicals. In the future, basic chemical raw materials that the industry needs will be produced by catalytic reactions from waste oils.

Due to the increases and fluctuations in oil prices in recent years, many countries have sought new alternatives to oil. As a result of these searches, they started to produce bio-diesel from vegetable oils as an alternative.

The aim of this study is to characterize linden seed oil, which has not been used in the oil industry recently, but whose production in large quantities will serve as a reference for the economy of the oilseeds in question, obtained by different methods and analyzing the fatty acid compositions (Kıllı & Beycioğlu, 2019).

2. Material and Methods

2.1. Materials

In this study, seeds from linden species available in Malatya Inonu University campus were collected and used. The collected seeds were separated from their shells and stored in lidded containers for experiments.



Figure 1. The hulls of the extracted linden seeds and the insides of the extracted linden seeds.

During the season, linden flowers were collected after they turned into seeds. Seeds are divided into shell and interior. After the inner seed was ground in agate air, it was sieved and analyzed when the grain size was 0.6-1.0 mm. Oil contents of linden seeds were determined according to AOCS standards. The amount of fat content of the samples was determined using the soxhlet extractor.

To determine the oil content, 10 g of ground seed is placed in the cartridge in the extractor section of the Soxhlet device. The chemical substance to be used as a solvent is placed in the glass balloon. With the help of the heater, this substance is evaporated. The evaporated solvent passes through the extraction column and reaches the reflux. The solvent condensed in the refrigerant comes back to the extraction column, dissolves the substance in the cartridge and returns to the glass balloon. This process is repeated continuously and the extraction is completed. After extraction, the solvent was removed from the flask, and the oil content of the seed was calculated by weighing the flask again. Hexane, acetone and ethyl alcohol were used as solvents in these process steps. Labota 4000-efficient Heidolph, USA was used as evaporator.

2.2. Chemicals

Chemicals used in the study: HCL(100314), NaOH (106462), KOH (105012), H₂SO₄ (100731), acetone (100014), hexane (107023), phenoltfalein (107233), ethyl Alcohol (818760) Merck grade was used.



Figure 2. Linden seed kernels ground in agate mortar for experiments.

2.3. Method

2.3.1. Analyzes on linden seeds

The following analyzes were made for the outer shell, oilseed, and oil-removed pulp of the test samples.

Ash determination

Ash determination was made according to TS-EN ISO 2171 standard. Approximately 1 g of ground linden seed was put into the crucible and placed in a muffle furnace at 650 °C. The door of the muffle furnace is opened and closed in order for oxygen to enter. The agate in the crucible stays in the oven until the linden seeds ground in the mortar are completely burned. After the product removed from the muffle furnace is cooled in the desiccator, it is put into the tared crucible. The same processes were repeated in the linden seed shell, the oiled linden inner seed.

$$K = 100 \times \frac{(WT - WC) \times 100}{(100 - M0) \times WS}$$
(1)

- K : Amount of ash by mass on a dry matter basis (%)
- M0 : Seed moisture (%).
- WC : Weight of the drying pan (g).
- WT : Total weight of pot and seed after processing (g).
- WS : Seed quantity (g).

Saponification number

The saponification number is the weight, in mg, of KOH required to saponify 0.5 g of oil. 0.5 g of ground seed is weighed into the balloon with a sensitivity of 0.001. 6.25 ml of 0.5 N ethanolic KOH solution is added on it. It is placed in the refrigerator for about 1 hour and boiled slowly by stirring from time to time. The balloon is taken from the refrigeration system and 4-5 drops of phenolphthalein are added to the hot soap solution and titrated with 0.5N H_2SO_4 solution until the red color of the phenol phthalein disappears.

Saponification number =
$$(Vk - V) \times 28.05 \text{ mg KOH} / \text{g oil}$$
 (2)

Vk : The amount of H₂SO₄ spent in the witness experiment, ml

V : The amount of H_2SO_4 spent for the sample, ml

m : Sample quantity, g

Chemicals used:

- Phenolphthalein solution 1% in ethanol
- 0.5 N KOH solution with ethanol
- $0.5 \text{ N H}_2\text{SO}_4 \text{ solution}$

Fatty acid components analysis

For fatty acid analysis, the oil obtained by soxhlet extraction was prepared for analysis by weighing 10 g for each type of oil into glass tubes and transferring it into a 15 ml plastic centrifuge tube with a lid. After adding 10 ml of n-hexane and closing the lid, it was shaken quickly. 0.5 ml of 2N methanolic KOH solution was added on the repeat and shaking was performed again. The supernatant was taken into the vial by injection, waiting until the supernatant clears (1-2 hours, in a dark environment). Our samples were made ready for the analyzer.

Fatty acid composition is determined by chromatographic devices and Gas Chromatography (GC-FID (Flame Ionization Detector) system is mainly preferred for this purpose (Nalçacı, 2020).

3. Results and Discussion

3.1. Oil extraction

After the seeds were collected, they were separated from their shells and the inner core yield was calculated. In the calculation, the inner core yield was 37% by weight, while the outer shell yield was 64% by weight. Fat percentages determined by Soxhlet extraction are given in Table 1.

| | Oil yields (%) | |
|-------|----------------|--|
| TM1H | 30 | |
| TMAC1 | 31 | |
| ТМЗН | 27.30 | |
| TMAC2 | 25 | |
| TMEt | 33 | |
| | | |

Table 1. Oil percentages of kernels extracted with different substances

TM1H: Seed oil extracted with hexane, TMAC1: Seed oil extracted with acetone, TM3H: Seed oil extracted with hexane, TMAC2: Seed oil extracted with acetone, TMEt: Seed oil extracted with Ethyl Alcohol

When Table 1 is examined, the highest oil yield was obtained by using ethyl alcohol as a solvent. The fact that the ethyl alcohol oil yield is higher than the others can be explained by the dissolution of the water in the structure in ethyl alcohol. As a matter of fact, acetone yield was higher than hexane. Low yield is an expected result since water does not dissolve in hexane. It is for this reason that hexane is used in the industry to obtain oil in oil seeds.

We can show that the oil yields of the hexane extract in the second oil forehead are lower than the previous one, as the reason for the seed to be moist.

3.2. Ash analyses

When Table 2 is examined, values close to each other were obtained in the results of the ash values of the pulp, which was degreased as a result of extraction. The reason for this supports the comments about the water passing to the solvent in oil yield. It is an expected result that the ash of the oily seed is low. Considering the oil yield, it supports the accuracy of the experimental results. Shell ash given as TMKK was lower than other biomass shells.

For the pH measurement of the ash, approximately 0.5 grams of ash was mixed with 25 ml of water and left to settle, and the measurement was taken with a pH meter in the clear part. The pH values of all ashes were measured by a pH meter, and a pH value of 12 and above indicates that these ashes are basic.

| | Ash (%) | | |
|--------|---------|--|--|
| TM1KUL | 5.91 | | |
| TM2KUL | 5.84 | | |
| TM3KUL | 5.85 | | |
| TM4KUL | 5.80 | | |
| TM5KUL | 5.50 | | |
| TMTK | 4.55 | | |
| ТМКК | 2.1 | | |
| | | | |

| Table 2 Ash | values and % | 6 ash ratios in | degreased linden | seeds linden | shell oily | inner core |
|--------------|--------------|-------------------|-------------------|---------------|------------|------------|
| Table 2. Ash | values and / | o asii ratios ili | ucgreased influen | secus, innuen | shon, ony | |

TMKUL: Ash of ground oil seed kernel, TMTK: Ground oil seed ash, TMKK: Ground bark ash

3.3. Saponification number in oils

By definition, saponification number is the mg weight of potassium hydroxide required to saponify 1g of oil. The saponification number of the oils and the chain lengths of the fatty acids, that is, the molecule weights, are inversely proportional. The saponification numbers of oils that are esters of long chain fatty acids are lower than the saponification numbers of oils that are esters of short chain fatty acids (Nehdi, 2011). For example, the saponification number in butter is high (between 210 and 235). The saponification number is 255 in coconut oil and 245 in palm oil. Apart from this, the saponification number of vegetable oils is generally below 200.

When Table 3 is examined, the saponification number of the oil obtained from the hexane extraction is quite low compared to the others. The high saponification number of oils obtained from acetone and ethyl alcohol can be explained by the increase in water and acidity in the environment.

Table 3. Results of saponification number in oils

| | Saponification number (mg KOH/ g oil) | |
|-------|--|--|
| TM1H | 157.08 | |
| TMAC1 | 241.43 | |
| ТМЗН | 196.35 | |
| TMAC2 | 252.45 | |
| TMEt | 232.48 | |

TM1H: Seed oil extracted with hexane, TMAC1: Seed oil extracted with acetone, TM3H: Seed oil extracted with hexane, TMAC2: Seed oil extracted with acetone, TMEt: Seed oil extracted with Ethyl Alcohol

Elemental analysis results of linden seeds and extracted oils are given in Table 4.

Table 4. Elemental analysis of linden seeds

| Sample Code | Carbon (%) | Hydrogen (%) | Nitrogen (%) | Sulfur (%) | Oxygen * (%) |
|-------------|---------------|-----------------|-----------------|---------------|-----------------|
| TM1 | 42.86 | 6.054 | 7.681 | 0.458 | 42.974 |
| TM2 | 43.47 | 5.922 | 3.122 | 0.123 | 47.363 |
| TM3 | 41.80 | 6.242 | 8.638 | 0.615 | 42.705 |
| TM4 | 41.06 | 5.663 | 4.887 | 0.307 | 48.083 |
| TM5 | 43.68 | 5.994 | 5.062 | 0.261 | 42.003 |
| TMEt | 75.71 | 11.36 | - | - | 12.93 |
| TMAC1 | 72.19 | 10.78 | - | - | 17.03 |
| TMAC2 | 73.33 | 10.81 | - | - | 15.86 |
| TM1H | 72.75 | 10.89 | - | - | 16.36 |
| TM3H | 69.01 | 10.24 | - | - | 20.75 |

* Calculated from the difference

TM1: Seeds extracted with hexane, TM2: Seeds extracted with acetone, TM3: Seeds extracted with hexane, TM4: Seeds extracted with acetone.

When Table 4 is examined, it is seen that the elemental compositions of the degreased inner cores are not different except for the nitrogen and sulfur values. Being rich in nitrogen shows that the seed is rich in amygdalin and protein. The sulfur value is close to all oil seeds and has an important place in both germination and nutrition. Differences in oxygen values are due to the difference in nitrogen and sulfur values. The results show that degreased linden seed can be used both as a food additive and as a feed additive in livestock.

The ash contents of the de-oiled kernels were almost similar in all samples, and this result showed that the ash compositions were the same.

When the elemental analysis results of the oils obtained from each extraction from the same table are examined; It is seen that carbon and hydrogen values in ethyl alcohol extraction are slightly higher than the others. Probably due to the solvent nature of ethyl alcohol, it can be explained by the passage of components soluble in ethyl alcohol from the composition other than oil. Elemental composition has similar values as a result of both extractions obtained for acetone and hexane. The absence of nitrogen and sulfur in all oils can be explained by the fact that they are in an organic form insoluble in oil and solvent. As a matter of fact, while amygdalin is soluble in water, it does not dissolve in oils.

3.4. Linden seed oil analysis

Fatty acid composition results of the oils obtained as a result of solid-liquid extraction of linden seeds by GC-MS are given in Table 6. When the table is examined, it is seen that linden seed oil is rich in oleic (27.070- 32.557) and lineleic (40.624-51.765) acids as a result of all extractions; shows that it is rich in unsaturated fatty acids. It is an important value that the stearic acid (1.945- 2.547) content is low compared to other seed oils. However, the palmitic acid value (9.775-12.750) is high, which can be explained by the ambient temperature in oil formation in plants. Since the linden flower seed usually transforms after mid-July, it is an expected result that palmitic acid will be high. The reason for the high palmitic acid grown in temperate regions is the ambient temperature (Samanci & Özkaynak, 2003).

| | TM1H | TMAC1 | TMAC2 | ТМ3Н |
|---|--------|--------|--------|--------|
| Fatty acid | %(w/w) | %(w/w) | %(w/w) | %(w/w) |
| C4:0 (butyric) | 0.097 | 0.257 | 0.362 | 0.238 |
| C6:0 (caproic) | 0.456 | 0.188 | 0.185 | 0.276 |
| C14:0 (myristic) | 0.310 | 0.419 | 0.348 | 0.358 |
| C15:1(cis-10-pentadecanoic) | 0.000 | 0.000 | 0.000 | 0.000 |
| C16:0 (palmitic) | 12.750 | 10.801 | 9.775 | 11.108 |
| C16:1(palmitolcic) | 0.135 | 0.124 | 0.116 | 0.125 |
| C17:0 (heptadecanoik) | 0.092 | 0.078 | 0.066 | 0.078 |
| C17:1 (cis-10-heptadecanoic) | 1.150 | 0.869 | 0.880 | 0.966 |
| C18:0 (stearic) | 2.547 | 1.945 | 2.050 | 2.180 |
| C18:1n9t (elaidic) | 2.329 | 2.811 | 3.083 | 2.741 |
| C18:1n9c (oleic) | 32.557 | 27.813 | 27.070 | 29.147 |
| C18:2n6t (linolelaidic) | 1.651 | 0.067 | 2.368 | 1.362 |
| C18:2n6c (linoleic) | 40.624 | 51.765 | 50.611 | 47.666 |
| C20:0 (arachidic) | 0.148 | 0.105 | 0.133 | 0.128 |
| C18:3n6 (a-linolenic) | 0.250 | 0.315 | 0.266 | 0.277 |
| C18:3n3 (g-linolenic) | 0.302 | 0.372 | 0.461 | 0.378 |
| C20:2 (cis-11,14-eicosadienoic) | 0.070 | 0.073 | 0.081 | 0.318 |
| C22:0 (behenic) | 0.058 | 0.037 | 0.048 | 0.047 |
| C23:0 (tricosanoic) | 0.000 | 0.000 | 0.046 | 0.015 |
| C24:0 (lignoceric) | 0.000 | 0.000 | 0.032 | 0.0011 |
| C20:5n3 (cis-5,8,11,14,17-eicosapentaoic) | 0.607 | 0.208 | 0.232 | 0.349 |
| C24:1 (nervonic) | 1.021 | 0.327 | 0.360 | 0.569 |
| C22:6n3 (cis-4,7,10,13,16,19-docosaeva) | 2.472 | 1.027 | 1.107 | 1.535 |

Table 5. Fatty acid analysis results

3.5. Linden seed SEM analysis

The results of the SEM analysis performed to determine the structural properties of the oily inner core and shell of the linden seed are given in Figure 3-4.

When the SEM images of the oil seed seeds in Figure 3 are examined, it is seen that the seed is in a very homogeneous structure. It is clearly seen that fats are in a cellular structure. With this structure, the homogeneous presence of oil in the inner seed protects the material from cold even at very low temperatures, as well as from extreme heat at high temperatures. It is also known that the oil in the seed has an important role in the rooting of the plant in sprouting.



Figure 3. SEM images of TM (In-ground oilseed).

When the SEM images of the ground shell given in Figure 4 are examined, the cellulosic structures are clearly seen. Hollow formations in the form of tubes are lignocellulosic structures, and their hollowness is the formations where mass and heat transfers take place between the outer part of the shell and the inner part.



Figure 4. SEM images of TMK (Ground Crust).

3.6. Linden seed FTIR analysis

The FTIR spectra of the internal seeds of which the structure remaining as a result of extraction with different solvents were removed are given in Figure 5. When the figure is examined, the structure in general is completely similar. The changes in the peak dimensions arise in terms of the homogeneity of the samples. The broad peak around 3250 cm⁻¹ belongs to the water hydroxyl and also belongs to the hydroxyl vibrations of the cellulosic structure. The double peak around 2942 cm⁻¹ belongs to the aliphatic C-H stresses, and these stresses belong to both fatty acids and cellulosic structures. The broad and sharp peak around 1078 cm⁻¹ belongs to inorganic M-O-M and C-O stresses. In organic materials containing inorganic structure, it usually occurs as a result of the structure of the ash.



Figure 5. FTIR plot of ground oily inner core (TM) and ground seed coat (TMK).



Figure 6. FTIR plot of degreased inner cores.

When the FTIR spectrum of the degreased inner cores in Figure 6 is examined, it is seen that the spectrum given for TM1 and TM3 of hexane extraction is similar. The absence of -C=O (carbonyl) stretching of the carboxyl group around 1700 cm⁻¹ indicates that all of the fatty acids were extracted with hexane. The broad peak seen around 2940 cm⁻¹ belongs to aliphatic -C-H structures and is attributed to cellulosic structures. The peak around 1078 cm⁻¹ belongs to the inorganic components and C-O-C structures of the degreased inner core. TM2 and TM4 belong to the inner core remaining from the acetone extraction and show that the fatty acid remained in the structure, albeit very little, in the TM2 sample. It is seen that TM 5 belongs to ethyl alcohol extraction and that the solvent in question has completely taken the oil from the inner seed.



Figure 7. FTIR plot of extracted oils.

When the FTIR spectra of the oils obtained with different solvents given in Figure 7 are examined, a very small –OH stretch peak around 3500 cm^{-1} is seen (Sim et al., 2012; Jayarambabu et al., 2014). This peak may be due to a very small amount of water in the oil. The two peaks at 2843 cm⁻¹ and 2917cm⁻¹ belong to aliphatic C-H stretches in fatty acids. The fact that both peaks are sharp and distinct also indicates that there are no other structures in the structure other than fatty acids. It is the stress belonging to the carbonyl (-C=O) group belonging to the carboxyl (-COOH) group in the sharp and distinct peak fatty acid at 1700 cm⁻¹. The partially broad peak around 1070 cm⁻¹ belongs to the C-O-stretch in the fatty acid structure.

In summary, the composition of linden seed oils obtained with different solvents is the same, which is an expected result.



Figure 8. FTIR plot of ash products.

The FTIR spectra of the ash values of the degreased inner cores are given in Figure 8 and when the figure is examined, it is seen that the inorganic composition of the ash is the same. The large and large peak around 1030 cm⁻¹ belongs to M-O-M structures in inorganic structure and is a very characteristic peak. The small sharp peak around 3284 cm⁻¹ belongs to the –OH stretch in the inorganic structure (Chen et al., 2010; Sim et al., 2012; Jayarambabu et al., 2014; Gawbah et al., 2017).

3.7. Linden seed XRD analysis

When Figure 9 is examined, it is seen that the ground oily inner core contains both amorphous and crystalline structural units. The XRD trace of the crystalline unit is around 23 20; While the ground oily inner core (TM) is seen in the structure of the ground seed coat (TMK), the absence of ground ash in the structure of the ground shell ash (TMKK) indicates that this structure, that is, the inorganic component(s) in the original structure, turns into an amorphous structure when the material is made. It probably belongs to ester structures made by inorganic components with organic components. It can be stated that the oily inner core shows 4 different amorphous structures, so the structure is mostly amorphous, the cellulosic structure of the shell is mostly crystalline, as well as showing at least 5 different amorphous structures. It can be stated that these amorphous units in cellulosic structure belong to soluble cellulosic structures, hemicellulosic structures and lignin and lignocellulosic structures (Pe et al., 2001).



Figure 9. XRD plot of ground oily inner core (TM), ground seed husk (TMK), and ground husk ash (TMKK).

4. Conclusion

The rapid increase in the world population, increasing urbanization, increasing technological innovations, the increasing importance of social life in the use of cosmetic products, the use of oil obtained from seeds in the fields of food, cosmetics, soap, etc. is increasing rapidly. In an environment where industrial oil seeds gain more importance, the food and cosmetics industry continues to search for new raw materials that will be a source of seed oils in the future. Evaluation of the seeds of all trees comes to the fore. Linden flower is an important herbal resource consumed in alternative therapy. However, if it is not collected, a product turns into a seed. In the literature study, it was determined that the study with linden seed oil was negligible. When the fatty acid content and yield and the characterization results are taken into account, it is concluded that linden seeds can be considered as an economical product in cases where the flowers cannot be collected. Therefore, it was necessary to carry out such a study. The results obtained at the end of this study;

The oils of the seeds collected from the linden trees in the Inonu Campus were obtained in a soxhlet mechanism with different solvents (hexane, acetone and ethyl alcohol). As a result of the analyzes made, the oil yield with acetone was 28% by weight, with hexane 28.65% and 33% ethyl alcohol values were determined. It has been determined that the oil yields are around 30% on average, and it has been concluded that the amount of unsaturated fatty acids is quite high, and it can even be considered as edible oil. It is clear that it can be used in every sector from soap making to cosmetics industry.

As a result of all extractions, it was determined that linden seed oil is rich in oleic (27.070-32.557) and linoleic (40.624-51.765) acids, its stearic acid (1.945- 2.547) content is lower than other seed oils, and its palmitic acid value is (9.775-12.750). In the fatty acid composition of linden tree seeds, the values of oleic acid: 27.442 for acetone, 30,852 for hexane, 10.955 for palmitic acid: acetone, 11.929 for hexane, 51.188 for linoleic acid: acetone, 44.145 for hexane were found. Soap number values are 176.72 for hexane in mg KOH/g fat unit; 246.94 for acetone; It was found as 232.48 for ethyl alcohol. Degreasing inner core ash determination values average 5.575%, bark ash was found to be 2.1% differently.

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