Efficiency of The Extraction Method for the Extracted Oil From the Local Pumpkin and Their Physiochemical Properties

Sheraz Zuher Karem[1] and Dawrin Hawar Mohammed[2]

[1,2]Food Technology Department, Agricultural Engineering Sciences College, University of Salahaddin, Erbil, Kurdistan Region, Iraq.

[1]E-mail: Sheraz.karem@su.edu.krd
[2]E-mail: darwin.mohammed@su.edu.krd

Abstract. Examining the oil output and the physiochemical effects of various extraction methods on pumpkin seeds was the primary goal of the research. We used n-hexan as a solvent in two separate oil extraction processes, cold press extraction (CPE) and soxhlet extraction (SE), and we choose pumpkin seeds for the job. In terms of oil yield, the SE technique achieved 64.07%, while the CPE approach came in at 36.33%. The physicochemical characteristics of the seed oils were discovered to be substantially affected by the oil extraction process, species, and the interaction between the two. Acidity, iodine, peroxide, saponification, density, refractive index, and viscosity were some of the physiochemical parameters measured for pumpkin oil. In terms of chemical tests, the Soxhlet extraction method outperformed CPE by 125, 3.02, and 1.03 iodine values, peroxide values, and acid values, respectively. On the other hand, CPE had a higher saponification number at 187.13, and SE had a lower one at 187.13. In terms of physical tests, the SE method outperformed CPE by 1.47, 0.916, and 76.4 viscosity, respectively. Both the oil yield and its physiochemical qualities were improved by the soxhlet extraction process, according to the results.

Keywords. Cold press extraction, Soxhlet extraction, Physical properties, Chemical properties.

1. Introduction

An herbaceous vine plant, pumpkins are members of the Cucurbita genus and the Cucurbitaceae family, which includes more than 119 other genera and 825 species. The pumpkin's flesh is versatile enough to be turned into dried fruits, pies, soups, liquids, cakes, and more; on the other hand, the seeds are roasted and eaten as flavourful snacks [1]. Food items made from pumpkin seeds are high in bioactive substances, sugar, fibre, minerals, fats, and protein. Particularly, lipids make up more than 30.0% of pumpkin seeds. The primary fatty acids found in these seeds are oleic acid (21–47%) and linoleic acid (36–61%), with oleic acid and stearic acid being the most abundant [2]. Recent years have seen a surge in interest in pumpkin seed oil (PSO) as a possible source of health benefits owing to its abundance of unsaturated fatty acids and micro-nutritive and antioxidant compounds, such as

phytosterol, tocopherols, squalene, phenolic acid, vitamins, and carotenoids [3]. Additional to its culinary uses, it has functional dietary properties that make it useful in the treatment and prevention of cardiovascular, cerebrovascular, and prostate illnesses [4]. Worldwide, PSO is known as a premium vegetable oil for use in food and medicine.

According to Dragoun et al, [5], PSO's nutritional value and functional activities were further dictated by its processing method, which significantly altered its composition and lipid companion content. Pretty pumpkins, or cucurbita spp., are well-known gourds that have dual purposes: eating and decorating. There are many practical and nutritious uses for the oil that is refined from their seeds. Essential fatty acids like oleic and linoleic acid are abundant in oil from certain plants. Right now, the US, Austria, Slovenia, Croatia, and Hungary are the ones that sell pumpkin oil [6]. Because oils are vulnerable to oxidative degradation, which shortens their shelf life, processing oils affects their nutritional capabilities and quality. Not only do physicochemical qualities (such as fatty acids, density, iodine value, saponification value, colour, and viscosity) impact oil's physical quality, but they also define the nutritional value. For oils to be commercialised while maintaining their quality, some factors must be considered [7].

Rumour has it that pumpkin seed extract and other plant components have antidiabetic, antihypercholesterolemia, anti-cancer, anti-tumor, antimutagenic, anti-oxidant, antibacterial, antihypertensive, anti-inflammatory, immunomodulatory, intestinal anti-parasitic, and antalgic characteristics [8]. Researching the physical, chemical, and microbiological properties of pumpkin seeds and oil is crucial since it will inform dietitians and doctors about the pumpkin's medicinal and nutritional uses [9]. The primary objective of this research is to evaluate the physiochemical characteristics of pumpkin seed oil that has been extracted using various methods.

2. Material and Method

2.1. Sample Collection
The pumpkin Produced in the Kurdistan Region and sourced straight from the farmer in Erbil. The pumpkin fruits were prepared by washing, drying, and chopping them by hand to extract the flesh from the seeds. We measured the weight of the seeds and averaged them out to get the percentage of seeds by weight of the whole pumpkin fruit.

2.2. Extraction Method
In this work, two distinct PSOs developed using CPE and SE. To begin, the LPP205, an automated cold (screw) press machine, was used to conduct the cold pressed extraction. The second step was to dry the pumpkin seeds in an oven at 105℃ for 24 hours. After that, they were crushed into a powder with a certain particle size. Then, they were extracted using a soxhlet apparatus. We transferred fifteen grammes of the material to a thimble. The 300 ml of n-hexane solvent was then added to the round bottom flask of the soxhlet apparatus, which is used for extractions in a Soxhlet apparatus and operates within a boiling range of 40-60 °C for a duration of 6 hours. Evaporation of the remaining solvent from the separated oil followed filtration under a vacuum and heating to 40 oC. We put the sealed oil in a dark container and kept it there until we could analyse it.

$$\% \text{ Yield} = \frac{W1-W2}{W1} \times 100 \tag{1}$$

W1= W eight of Sample (g)
W2 = Weight of remaining sample (g).

2.3. Physicochemical Characterization of Pumpkin Seed Oil
The following physicochemical characteristics of the oils that were extracted were assessed according to the official procedures set out by the American Oil Chemists' Society [10]:

2.3.1. Chemical Properties

2.3.1.1. Iodine Value

The number of milligrammes of iodine that one gramme of oil absorbs is called its iodine value (IV). Intravenous fatty acid testing determines the percentage of fat that is unsaturated. In order to break the unsaturated fatty acids' double bonds, a halogen is introduced into the process; the iodine number is a measure of the quantity of halogen injected. The overall unsaturation content of a given oil or fat is indicated by its IV [11]. The oil extracted from pumpkin seeds by Dissolve 18.2 grammes of iodine in 1 litre of glacial acetic acid. Bring the halogen level up by adding 3 millilitres of bromine water. Hansus solution may be made by mixing 1 gramme of pumpkin oil with half a millilitre of chloroform solvent. The next step is to add five millilitres of distilled water and half a millilitre of potassium iodide to the mixture. Then, after adding two or three drops of starch, the solution is let to settle for half an hour [10]. Next, it is estimated by titrating with a sodium thiosulphate solution. We used the following equation to get the iodine value:

$$\text{Iodine Value} = \frac{[(B-S)\times N\times 126.9]}{\text{weight of sample(g)}} \tag{2}$$

Where:
The molecular weight of iodine is 126.9, and N is the normality of the Na2S2O3 solu-tion. S= millilitres of titrant with sample and B= millilitres of titrant without blank.

2.3.1.2. Peroxide Value

An early stage oxidation index for lipids is the peroxide value. The PV of lipids and oils is a key metric for evaluating the quality of storage oil. The maximum allowable peroxide value for oil is 10 milliequivalents [12]. Pumpkin oil samples' peroxide potency. To make a homogenous mixture, add 1 gramme of pumpkin oil and stir for 1 minute in a dark place after adding 25 millilitres of glacial acetic acid (16.6 millilitres), chloroform (8.4 millilitres), and KI (1 gramme). 35% distilled water, 2-3 drops of starch for colour, and lastly, titratable sodium thiosulphate. The peroxide oxygen equivalents per kilogramme of oil were measured in milligrammes (meq/kg).

$$\text{Peroxide Value} = \frac{[(S-B)*N]\times 1000}{\text{weight of sample(g)}} \tag{3}$$

Where:
S= Volume of titrant (ml) with sample, B= Volume of titrant (ml) with blank, $N=Normality$ of sodium thiosulfate

2.3.1.3. Acid Value

The quantity of potassium hydroxide needed to neutralise the free fatty acids in one gramme of oil or fat sample is known as the acid value (AV) and is measured in milligrammes. The amount of glyceride degradation in oils caused by lipase may be determined by AV [11]. collected pumpkin oil samples and determined their acidity using the method outlined in [10]. Before titrating with 2-3 drops of phenolphthalein indicator, a 0.1 N potassium hydroxide solution was made by dissolving 1g of oil sample in a 50:50 mixture of ethanol and ether. The acid value and free fatty acid quantity are both quantified by this equation.

$$\text{Acid Value} = \frac{\text{VofKOH}\times N\times 56}{\text{weight of sample(g)}} \tag{4}$$

Where
V: Voltage of the potassium hydroxide titrant (ml),
N: normality of the potassium hydroxide (N),
56: molecular weight of potassium hydroxide (56 g/mol),

2.3.1.4. Saponification value

The acid produced by fully hydrolysing one gramme of oil sample would be neutralised by this amount of potassium hydroxide (KOH), in milligrammes (mg). I pipetted 25 millilitres of the alcoholic potassium hydroxide solution into a conical flask containing two grammes (2 g) of oil. A reflux condenser was subjected to hourly shaking while being heated on a water bath that was boiling. Titrated when hot with the standard hydrochloric acid (a ml), a millilitre (1 ml) of phenolphthalein solution served as the indicator. A measurement of blank volume (b ml) was taken.

2.3.2. Physical Properties

2.3.2.1. Refractive Index

Following the procedure outlined by AOAC [13], the refractive index was calculated. With two to three drops of extracted pumpkin oil per sample, the samples were placed on the sample position of the refractometer (Refractometer, ARAGO, Digital Thermo meter, KOICA). All samples had their refractive indices measured at 25 °C.

2.3.2.2. Density

What this means is that for every volume of oil at 20°C, the mass in air is proportional to the mass of water at 25°C in the same volume. Fill a 50 mL pycnometer with distilled water and set it on a water bath to 25°C after it had cleaned, dried, and weighed. After drying it off, the density bottle was weighed. The process for determining the oil's weight at 25°C was same.

2.3.2.3. Viscosity

In accordance with [14], a digital rotary viscometer (Viscometer, NDJ-8S, Digital Rotary Viscometer, China) was used to test the viscosity of each sample at 25 °C.

2.4. Statistical Analysis

Statistical analysis was performed on the data obtained using the SPSS software (SPSS software, 2019). At a significance threshold of ($p < 0.05$), we compared the averages using Duncan's multiple range tests.

3. Result and Discussion

3.1. Oil Yield

The organoleptic qualities and traits of edible oils are significantly influenced by their physicochemical properties. As a result, understanding the many variables that affect these qualities is intriguing. Figure 1 displays the effects on the physicochemical parameters of the pumpkin oils of the two extraction methods (CPE and SE) and their interaction ($p < 0.05$). Almost every property was significantly affected by the variables that were researched and how they interacted with one another. The oil yield was 64.07% when extracted using the SE technique, whereas it was 36.03% when extracted using the CPE method. The cellular membranes found in seeds are the primary obstacle to oil extraction using the CPE technique. When compared to other extraction procedures, SE produced the highest oil output because it quickly breaks down cellular membranes, making them porous and improving oil extraction [15]. The findings published by [16] are well-supported by the results of oil yield in various techniques.
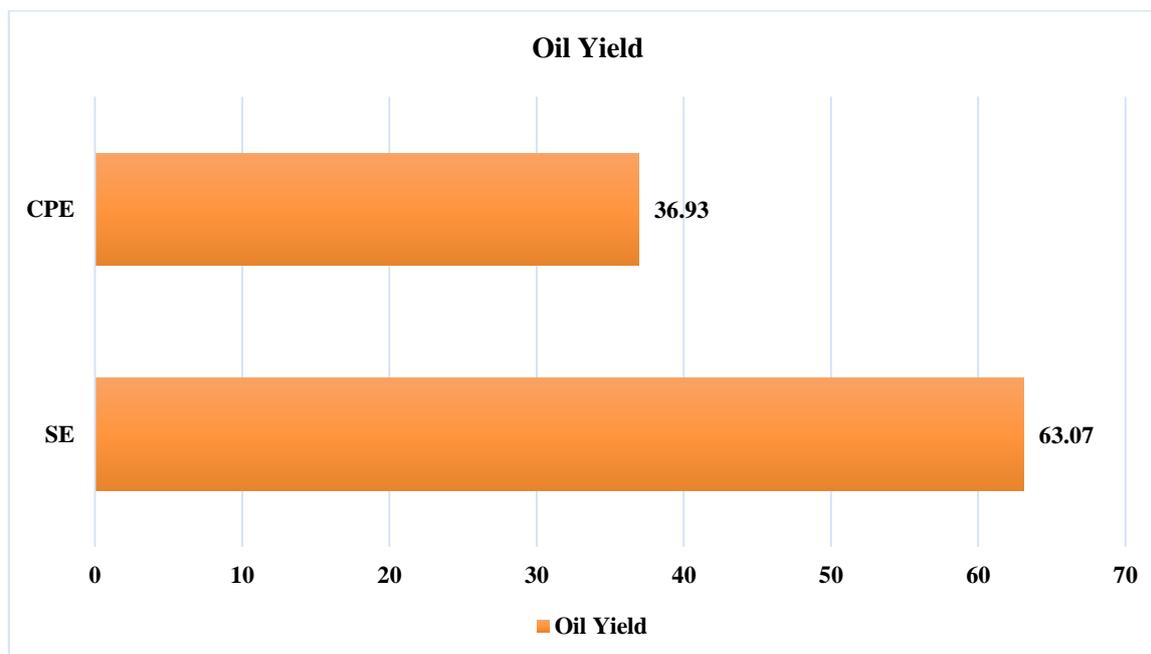
Figure 1. The effect of different extraction method on oil yield.

3.2. Physicochemical Properties

3.2.1. Chemical Properties

Table 1. Chemical properties of CPE and SE and oil.

Tests	Cold Press Extraction	Soxhlet Extraction
Iodine Value	113 ± 1.03^b	125 ± 1.178^a
Peroxide Value	1.80 ± 0.12^b	3.02 ± 0.08^a
Acid Value	0.82 ± 0.01^b	1.03 ± 0.03^a
Saponification Number	187.13 ± 1.03^a	181.09 ± 0.082^b

Mean $\pm$ SD with significant difference ($p \leq 0.05$).

3.2.1.1. Iodine Value
Table 1 displays the results of the two extraction methods and iodine value levels. discovered that the iodine value varied from $113 \pm 1.03b$ g to $125 \pm 1.178a$ g, with the lowest value seen in CPE oil and the greatest value in SE oil. Higher linoleic and oleic acid concentrations in an oil sample give rise to a higher iodine value, which in turn indicates a higher degree of unsaturation; yet, oils with a high iodine value are more likely to rancidify and need special storage conditions to preserve their quality [17].

3.2.1.2. Peroxide Value
An essential metric for edible oil quality monitoring, peroxide value (PV) reveals the degree of primary oxidation in the oil. The values of PV, which range from 1.80 to 3.02 meq O2/kg oil, demonstrated a significant difference ($p < 0.05$) between the two techniques of oil extraction. The use of solvent, heat, and oxygen during extraction may have contributed to the highest peroxide value (3.02 meq O2/kg oil) for SE oil. This increased oxidation may impact the oil's flavour, fragrance, and shelf life [18]. At its lowest point, in 1.80 With a low PV value, you may be sure that the product has excellent nutritional content, flavour, and quality since it is very resistant to oxidation [19].

3.2.1.3. Acid Value

As seen in Table 1, the acid value (AV), which indicates the rancidity of oil by measuring the presence of free fatty acids in oil, differed considerably across all the oils extracted using CPE and SE techniques. Oil extracted using the SE technique had a higher AV value (1.03 mg KOH/g oil). This is likely due to the fact that the extraction operation was run at 60 °C for a longer period of time, which hydrolyses TAG molecules and produces free fatty acids. With the lowest AV value (0.82 mg KOH/g oil) of the available methods, the CPE process is superior for producing high-quality oil. The AV results were quite consistent with those of Jafari *et al.* [20].

3.2.1.4. Saponification Value

Lipid triglyceride purity and molecular weight are reflected in the saponification value. Results for PSOs extracted using the SE method range from 181.09 to 187.13 on the saponification scale; a higher number indicates the presence of shorter chain fatty acids, which necessitate more potassium hydroxide for saponification; and the CPE method yields the highest number. Consistent with previous reports, this study found that compared to CP oil, MP vegetable oil had better acid, peroxide, saponification, moisture, and iodine values [21].

3.2.2. Physical Properties

Table 2. Physical properties of CPE and SE of pumpkin seed oil.

Tests	Cold Press Extraction	Soxhlet Extraction
Refractive Index	1.4676 ± 0.01^b	1.47 ± 0.00^a
Density	0.897 ± 0.03^b	0.916 ± 0.02^a
Viscosity	70.8 ± 0.01^b	76.4 ± 0.02^a

Mean ± SD with significant difference ($p \leq 0.05$).

3.2.2.1. Refractive Index (RI)

The optical metric known as the refractive index (RI) examines the light beams as they pass through a material. As the quantity of conjugated fatty acids produced by thermal deterioration of oil during frying or auto oxidation of oil grows, the value of RI also increases. There was no significant difference ($p < 0.05$) in the refractive index values of the oil samples obtained from any of the three procedures. The RI values for the SE and CPE oil samples were 1.47 and 1.4676, respectively. Our refractive index result falls within the range of values reported by the Codex Standard for physical and chemical indices. There is a strong correlation between the molecular mass or relative density of an oil and its refractive index value. The published refractive index values of 1.4662 and 1.465 for pumpkin seed oil, respectively, by Ardabili *et al.* [22] and Tsaknis *et al.* [23], are quite near to this number.

3.2.2.2. Density

Engineering design for oil production, fluid transportation, surface processing, and material balance calculations all rely on the crucial feature of oil density at the bubble point, which is also necessary in reservoir engineering calculations. At a certain pressure and temperature, the mass per unit volume is called the oil density [24]. With a density of 0.916 and a CPE density of 0.897, respectively, the SE extraction technique has a greater density than the CPE approach. Density is a measure of the number of molecules per unit volume. Dissimilarities in oxidation state, molar mass, and fatty acid content, together with potential contaminants in the oils [25].

3.2.2.3. Viscosity

Shear factor The amount of internal friction in an oil that makes it resistant to flow is called its viscosity. It is a crucial component in lubricants as it determines the oil's flow resistance. The shear stress-to-flow-rate ratio is another way to describe viscosity [26]. The SE extraction has the highest

viscosity at 78.4 while the CPE has the lowest at 70.8. Quickly flowing oils are those with a low viscosity [27].

Conclusion

Beginning with oil yield, the SE extraction method outperforms the CPE method. This is due to the fact that the SE has a higher oil yield. Moving on to physiochemical properties, the SE method outperforms the CPE method with the exception of saponification number, where the CPE method comes in at 187.13 and the SE method comes in at 181.09.

References

[1] Hussain, A., Kausar, T., Sehar, S., Sarwar, A., Ashraf, A. H., Jamil, M. A., ... & Majeed, M. A. (2022). A Comprehensive review of functional ingredients, especially bioactive compounds present in pumpkin peel, flesh and seeds, and their health benefits. Food Chemistry Advances, 1, 100067. doi.org/10.1016/j.focha.2022.100067

[2] Habib, A., Biswas, S., Siddique, A. H., Manirujjaman, M., Uddin, B., Hasan, S., ... & Asaduzzaman, M. (2015). Nutritional and lipid composition analysis of pumpkin seed (Cucurbita maxima Linn.). J Nutr Food Sci, 5(4), 374. DOI 10.4172/2155-9600.1000374

[3] Kulczyński, B., & Gramza-Michałowska, A. (2019). The profile of carotenoids and other bioactive molecules in various pumpkin fruits (Cucurbita maxima Duchesne) cultivars. Molecules, 24(18), 3212. https://doi.org/10.3390/molecules24183212

[4] Šamec, D., Loizzo, M. R., Gortzi, O., Çankaya, İ. T., Tundis, R., Suntar, İ., ... & Nabavi, S. M. (2022). The potential of pumpkin seed oil as a functional food—A comprehensive review of chemical composition, health benefits, and safety. Comprehensive reviews in food science and food safety, 21(5), 4422-4446. https://doi.org/10.1111/1541-4337.13013

[5] Dragoun, M., Klausová, K., Šimicová, P., Honzíková, T., Stejskal, J., Navrátilová, K., ... & Kyselka, J. (2022). Formation of previously undescribed δ7-phytosterol oxidation products and tocopherylquinone adducts in pumpkin seed oil during roasting, screw-pressing, and simulated culinary processing at elevated temperatures. Journal of Agricultural and Food Chemistry, 70(37), 11689-11703. https://pubs.acs.org/doi/10.1021/acs.jafc.2c03292?goto=supporting-info

[6] Rezig, L., Chouaibi, M., Msaada, K., & Hamdi, S. (2012). Chemical composition and profile characterisation of pumpkin (Cucurbita maxima) seed oil. Industrial Crops and Products, 37(1), 82-87. https://doi.org/10.1016/j.indcrop.2011.12.004

[7] Xiang, C., Xu, Z., Liu, J., Li, T., Yang, Z., & Ding, C. (2017). Quality, composition, and antioxidant activity of virgin olive oil from introduced varieties at Liangshan. LWT, 78, 226-234. https://doi.org/10.1016/j.lwt.2016.12.029

[8] Aremu, M. O., Ibrahim, H. and Bamidele, T. O. (2015). Physicochemical characteristics of the oils extracted from some Nigerian plant foods: A review. Chemical and Process Engineering Research, 32, 36 – 52. https://doi.org/10.3923/jas.2007.835.840

[9] Siano, F., Straccia, M. C., Paolucci, M., Fasulo, G., Boscaino, F. and Volpe, M. G. (2016). Physico-chemical properties and fatty acid composition of pomegranate, cherry and pumpkin seed oils. J. Sci. Food Agric., 96, 1730–1735. http://dx.doi.org/10.1002/jsfa.7279

[10] AOAC (Association of Official Analytical Chemists). (2000). Official Methods of Analysis (17th ed.). AOAC International.

[11] Babagana, G., Bamidele, S. S. and Idris, M. B. (2011). Characterization and Composition of Balanites aegyptiaca seed oil and its potential as biodiesel feed stock in Nigeria. J. of Applied Phytotech. in Envtal Sanitation, 1, 29–35. https://doi.org/10.5012/jkcs.2011.55.4.680

[12] Basuny, M. M., Arafat, M. and Ahmed, A. A. (2012). Vacuum frying: An alternative to obtain high quality potato chips and fried oil. Banats. J. Biotechnol., 3, 22–30. http://dx.doi.org/10.1007/s12393-011-9037-5

[13] AOAC (Association of Official Analytical Chemists). (2005). Official Methods of Analysis (18th ed.). AOAC International.

[14] Emil, A., Yaakob, Z., Satheesh Kumar, M.N., Jahim, J.M. & Salimon, J. (2010) Comparative evaluation of physicochemical properties of jatropha seed oil from Malaysia, Indonesia and Thailand. Journal of the American Oil Chemists' Society. 87 (6). 689-695. http://dx.doi.org/10.1007/s11746-009-1537-6

[15] Chouaibi, M., Rezig, L., Hamdi, S., & Ferrari, G. (2019). Chemical characteristics and compositions of red pepper seed oils extracted by different methods. Industrial Crops and Products, 128, 363–370. http://dx.doi.org/10.1016/j.indcrop.2018.11.030

[16] Seymen, M., Uslu, N., Turkmen, O., Al Juhaimi, F., & Ozcan, M. M. (2016). Chemical compositions and mineral contents of some hull-less pumpkin seed and oils. Journal of the Americal Oil Chemists Society, 93, 1095–1099. http://dx.doi.org/10.1007/s11746-016-2850-5

[17] Ozcan, M. M., Ghafoor, K., Al Juhaimi, F., Ahmed, I. A. M., & Babiker, E. E. (2019). Effect of cold-press and soxhlet extraction on fatty acids, tocopherols and sterol contents of the Moringa seed oils. South African Journal of Botany, 124, 333–337. https://doi.org/10.1016/j.sajb.2019.05.010

[18] Ahmed, I. A. M., Al- Juhaimi, F. Y., Ozcan, M. M., Osman, M. A., Gassem, M. A., & Salih, H. A. A. (2019). Effects of cold- press and soxhlet extraction systems on antioxidant activity, total phenol content, fatty acids and tocopherol content of walnut kernel oils. Journal of Oleo Science, 68, 167–173. https://doi.org/10.5650/jos.ess18141

[19] Haile, M., Duguma, H. T., Chameno, G., & Kuyu, C. G. (2019). Effects of location and extraction solvent on physico chemical properties of Moringa stenopetala seed oil. Heliyon, 5(11). https://doi.org/10.1016/j.heliyon.2019.e02781

[20] Jafari, M., Goli, S. A. H., & Rahimmalek, M. (2012). The chemical composition of the seeds of Iranian pumpkin cultivars and physicochemical characteristics of the oil extract. European Journal of Lipid Science and Technology, 114, 161–167. https://doi.org/10.1002/ejlt.201100102

[21] Ghazani, S. M., García-Llatas, G., & Marangoni, A. G. (2014). Micronutrient content of cold-pressed, hot-pressed, solvent extracted and RBD canola oil: Implications for nutrition and quality. European Journal of Lipid Science and Technology, 116(4), 380-387. https://doi.org/10.1002/ejlt.201300288

[22] Ardabili, A. G., Farhoosh, R. and Haddad Khodaparast, M. H. (2011). Chemical composition and physicochemical properties of pumpkin seeds (Cucurbita pepo Subsp. pepo Var. Styriaka) grown in Iran. J. Agr. Sci. Tech., 13, 1053–1063 http://jast.modares.ac.ir/article-23-9897-en.html

[23] Tsaknis, J., Lalas, S. and Lazos, E. S. (1997). Characterization of crude and purified pumpkin seed oil. Grasasy Aceites, 48, 267– 272. https://doi.org/10.3989/gya.1997.v48.i5.802

[24] Hamitri-Guerfi, F., Ouahrani, S., Benbouriche, A., Bey, M. B., Boulekbache-Makhlouf, L., & Madani, K. (2020). Impact of the extraction method on physico-chemical properties, phytochemicals, and biological activity of pumpkin seeds oil. The Annals of the University Dunarea de Jos of Galati. Fascicle VI-Food Technology, 44(1), 82-103. https://doi.org/10.35219/foodtechnology.2020.1.05

[25] Aktaş, N., Gerçekaslan, K. E., & Uzlaşır, T. (2018). The effect of some pre-roasting treatments on quality characteristics of pumpkin seed oil. OCL, 25(3), A301. https://doi.org/10.1051/ocl/2018025

[26] Bhattad, A. (2023). Review on viscosity measurement: devices, methods and models. Journal of Thermal Analysis and Calorimetry, 148(14), 6527-6543. https://doi.org/10.1007/s10973-023-12214-0

[27] Yang, X., Zhang, D., Ye, Y., & Zhao, Y. (2022). Recent advances in multifunctional fluorescent probes for viscosity and analytes. Coordination Chemistry Reviews, 453, 214336. https://doi.org/10.1016/j.ccr.2021.214336.