A new aqueous method for recovering high quality oil and deoiled meal from pecan kernels

Shuting Fu^1 and Wenbiao Wu^2

¹Affiliation not available ²Southwest University

February 22, 2024

Abstract

The extraction of oil by a new aqueous method has been promoted because it is environmentally friendly, safe and economical of cost. A new aqueous method using 1.4:10.0 water-to-pecan kernel slurry ratio was developed, which recovered 97.73% of oils from the kernel containing 70.47% crude oil content. The method had a higher oil recovery as compared to enzyme-assisted extraction or solvent extraction or cold pressing in terms of producing oil for safe consumption. The method recovered oils with 0.47 mg KOH/g acid value and 0.34 mmol/kg peroxide value which were identical to that obtained by enzyme-assisted aqueous method and lower than that obtained by solvent extraction. The de-oiled pecan meal obtained by the new aqueous method only contained 5.14% residual oils, which was edible since no harmful chemical was added during oil extraction. No waste water was produced during the aqueous extraction of oils.



Introduction

Pecan (*Carya illinoinensis*) is a woody oil crop of the Juglandaceae. The advantages of pecan kernel have been widely reported in the literature (Lv and Liu, 2005; Wang et al., 1998; Wang et al., 2012). Pecan kernel

contains up to 65-75% oil and the dry matter remaining is rich in proteins. Pecan oil was considered a healthy specialty gourmet product (Cockerham et al., 2012; Al Juhaimi et al., 2018). The functional advantages of de-oiled pecan meal have also been reported in the literature (Marchetti et al., 2017). Furthermore, the advantages of vegetable proteins over animal proteins have been stated in the literature (Asgar et al., 2010).

A method which is able to simultaneously produce high quality oil and de-oiled meal is generally preferable to other methods for processing pecan kernels. Solvent extraction has the following disadvantages: 1) not environmental friendly, 2) explosion and fire risk, 3) not safe for consumption before the refinement of the crude oils extracted, 4) costly to operate, and 5) adverse effect of solvent on human health (Tu et al., 2017; Latif and Anwar, 2009; Environmental Protection Agency, 1999) though it can efficiently produce high quality oil and de-oiled meal on a commercial scale (Johnson, 2000; Lamsal et al., 2006; Pare et al., 2014). Conventional high temperature pressing produces dark de-oiled meal with high residual oil content (inadequate recovery of oils) and denatured proteins lacking functional properties and having limited application to the food industry. Enzyme-assisted aqueous methods (EAAM) using large amounts of water may simultaneously produce high quality oil and hydrolyzed proteins (Sharma and Gupta, 2004; Sharma and Gupta, 2006; Zhang et al., 2011; Liu et al., 2017). The mechanism of this method for the extracting oil is associated with the full dispersion or solubilization of proteins, free amino acids, free fatty acids, phospholipids, polyphenols, alkaloids, etc. which have both hydrophilic and hydrophobic groups to form an emulsion. However, four main disadvantages of this method are reported: 1) low oil recovery rate or even no free oil produced because of formation of a severe emulsion during the extraction process, 2) difficulty of treating large amounts of waste water generated with high cost, 3) removal of water from de-oiled meal is costly, and 4) use of expensive enzyme (Li et al., 2016; Ravber et al., 2015; Zhang et al., 2011; Jiang, 2010; Hanmoungjai et al., 2000).

The aim of this study was to develop a new aqueous method (NAM) for processing pecan kernels. The NAM established was compared with enzyme-assisted aqueous procedure using large quantities of water, and with hexane extraction in respect to oil recovery (OR) and the quality of de-oiled meal or oil obtained.

Materials and Methods

Materials

Pecan nuts were bought from Longgan Hongshun Food Plant, Linan, Zhejiang, China. Their kernels were obtained by hand shelling.

Extraction Process Design by Single Factor Experiments

General Procedure in All Experiments

The process flow diagram of producing oils and deoiled meal from pecan kernels by water is shown in Fig. 1. The pecan kernels were baked for certain minutes (variable studied, 0 to 10 minutes) under a certain temperature (variable studied, 24 to 120). They were then ground to slurry passed through a sieve (made by Hebei Jiufeng Screen Metal Products Factory, Hebei, China) with a certain mesh (variable studied, 80 to 300 meshes) with a certain grinding rod by hands. The slurry (10.00 g) was weighed into a centrifuge tube (20 ml) and mixed with a certain amount (variable studied, 0 to 1.60 mL) of deionized water. The mixture was agitated using a stainless steel rod for certain minutes (variable studied, 0 to 60 minutes) under a certain temperature (variable studied, 24 to 60) at 60 rpm. Free oil produced was collected by centrifugation at 1435 g for 30 minutes at room temperature three times (the Model 80-2 Electric Centrifuge made by Jintan Medical Equipment Factory, Jintai, China) followed by spiral cold expressing (the BG-03 cold screw expeller made by Aibang Agricultural and Horticultural Machinery Factory, Shanghai, China). Each sample was repeated three times.

Optimization of Extraction Conditions via Single Factor Experimentation

The conditions of aqueous extraction were optimized via single factor experimentation under the condition of that the general procedure just mentioned above in "2.2.1." was followed, using OR as an indicator. All

the extraction conditions studied are indicated in Table 1.

The oil content of de-oiled pecan meal vacuum-dried to the constant weight at 50 was measured according to the Soxhlet method (Chinese National Standard, 2008). The calculation of OR was carried out according to follows:

OR (%) = $(X_1-X_2)/X_1 \times 100\%$

In this formula, X_1 (g) means quantity of crude oils extracted from the pecan kernel slurry (i.e. 10.00 g \times fraction of crude oil from the pecan kernel slurry (dry basis)), while X_2 (g) means quantity of crude oil remaining in the de-oiled pecan meal (i.e. amount of de-oiled pecan meal (g, dry basis) \times its fraction of oil).

Further Optimization by Response Surface Method

Four conditions (i.e. baking temperature, sieve mesh, quantity of addition of water as well as time of agitation) were selected for optimization by using response surface method according to the results of the single factor experimentation previously carried out. Investigation into cross-effects of multi-factors on OR and further optimization of operating conditions were carried out by using Box-Behnken's central combined experimentation design.

Solvent Extraction of Oil

The pecan kernels were baked at 95 for 8 min, cooled, pulverized for 30 s using a BJ-150 pulverizer (made by Baijie Jingdong Company, China), ground to pass through a 100-mesh sieve with a ceramic grinding rod by hands, and extracted for 10 h at 85 in a water bath by Soxhlet extractor with n-hexane as solvent. The extract was evaporated in a rotary evaporator until n-hexane was completely removed. Free oil was vacuum dried at 50 to a constant weight. Oil content in de-oiled meal was determined. Oil extraction efficiency (OEE) was calculated by the method similar to that described in "2.2.2" for calculating OR (= OEE) of NAM. Crude oil obtained was refined by method similar to that optimized by Ma et al. (2017). OR of solvent extraction (SE) was calculated by: [total refined oil obtained (g)/total crude oil in original pecan kernels (g)] x 100.

Analytical Methods

Content of crude oil in each sample was analyzed by referring to GB/T 14488.1-2008 (Chinese National Standard). After each sample (containing < 10% water) was extracted by using petroleum ether for 4 hours in a Bolton-Williams direct drop extractor, the sample-containing filter cartridge was taken out and the absorbed solvent was removed by an evaporator. The first extracted sample was poured out from the filter cartridge and crushed for 7 minutes. The crushed sample was quantitatively transferred to the filter cartridge and then extracted for 4 hours. This step was repeated once. Finally, the sample was weighed after the complete removal of solvent and crude oil content was calculated.

Acid value (AV) of each sample was analyzed by referring to GB/T 5530-2005 (Chinese National Standard). Each sample was dissolved in neutralized solvent and then titrated by using KOH solution.

Peroxide value (PV) of each sample was analyzed by referring to GB/T 5538-2005 (Chinese National Standard). Each sample in acetic isooctane reacted with potassium iodide. The iodine formed was titrated by using sodium thiosulfate solution. PV was finally calculated.

Statistical Analysis

One-way analysis of variance was used to analyze experimental results. Student's t-test was used to estimate the significance of difference of paired data. Microsoft Office Excel was used to calculate t-value. Design Expert 8.0.6 software was used for response surface analysis. SAS V8.0 was used to establish multifactorial regression model.

Results and Discussion

The content of crude oil of the pecan kernel slurry investigated was 70.43%. All data in terms of OR were calculated on the basis of this result.

Extraction Process Design by Single Factor Experiments

E flect of Water Added on Oil Recovery

The effect of water added on OR is shown in Fig. 2. The OR was only 37% without the addition of water. It gradually increased as the quantity of added water was augmented from 0.00 to 1.40 mL, but failed to increase further when water was increased to 1.60 ml. Among all of the water dosages investigated, 1.40 mL per 10.00 g slurry resulted in the highest OR.

Technically, the water added is necessary for extracting pecan oil and its amount of addition was found to be a critical factor of obtaining a reasonably high OR. The separation of oils by the addition of water may be involved with the mechanism that the cohesion work of oils and solid particles overcomes the adhesion work of oils to the solid particle surface. When the pecan kernels were converted to a slurry by grinding, the formation of a continuous oily phase occured because of release of free oils. Such compounds as proteins, phospholipids, and free fatty acids compose many small solid particles dispersed which have hydrophobic surfaces covered by oils. At this state, the work of adhering to the solid particle surface by oils is larger than that of cohesion of oils plus that of centrifugation force (1435 g). When water is added, it binds to the hydrophilic groups the surface of the small solid particles facilitated by mechanical agitation and therefore hydrogen bonds are formed. A rigid, sticky and particle is formed because of the aggregation of all the hydrophilic groups and water driven by hydrogen bond. At this state, inter surface tension is increased. The work of adhering to the solid particle surface by oils is less than that of cohesion of oils so that free oils are released.

Effect of Time and Temperature of Baking on Oil Recovery

The effect of time of baking on OR is shown in Fig. 2. The OR was only 77% without baking. It gradually increased as the baking time was increased from 0.0 to 8 minutes. However, it dramatically decreased when the baking time was further increased to 10 minutes. Among the baking time studied, baking for 8 minutes was the most suitable for recovering oil from the pecan kernel slurry. Therefore, proper baking time will increase the OR though baking for too long adversely affected the OR.

The effect of temperature of baking on OR is also shown in Fig. 2. The OR was only 77% without baking. It gradually increased as the baking temperature was increased from 24 to 95. However, it decreased when the baking temperature was further increased to 120. Among all the baking temperature investigated, baking at 95 resulted in the highest OR.

Baking the pecan kernels ahead of grinding is necessary for deactivating lipase and removing water. Lipase deactivation is able to prevent neutral oils from hydrolysis during grinding and extraction by water. Undesirable losses of neutral oil can be reduced if its hydrolysis is prevented so that di- or mono-glycerides, glycerols and free fatty acids formation can be decreased. This step may also be important for avoiding emulsion during aqueous extraction or grinding. This should also be a critical factor of obtaining the best OR. Furthermore, proper baking may facilitate the disruption of cell wall by grinding. However, increases in baking time or temperature might elevate the extent of protein denaturation, which might decrease OR because of an increase in oil absorption.

Effect of Saline Concentration on Oil Recovery

The effect of saline concentration on the OR is shown in Fig. 2. The OR was 97.73% when water was added without the addition of salt. It gradually decreased as the saline concentration was increased from 0% to 10%. Therefore, the addition of salt adversely affected the OR. The reason for obtaining this result is unknown. Salt may compete with hydrophilic compounds for water so that their hydration is not enough for releasing oils.

Effect of Amount of Brine Added on Oil Recovery

Fig. 3 indicates the impact of added brine on OR. It was only 37% without the addition of brine (aqueous NaCl solution). It gradually increased as the quantity of added brine raised from 0.00 to 1.80 mL. However, it decreased when the amount of brine added was further increased to 2.00 mL. It should be noted the highest OR obtained by the addition of 1.80 mL brine (93.82%) was lower than that obtained by the addition of only water (97.73%). This experiment again proved that salt did not improve the OR.

Effect of Temperature and Time of Agitation on Oil Recovery

The effect of temperature during agitation on OR is shown in Fig. 3. The OR was maximum (97.73%) when the agitation was undertaken at room temperature (24). It gradually decreased as the agitation temperature was increased from 24 to 60. Therefore, an increase in agitation temperature adversely affected OR.

The effect of time of agitation on OR is shown in Fig. 3. The OR was 65% without stirring. The increase in the OR was very significant when the agitation time was raised from 0 to 30 minutes. As the agitation time continued to increase, the OR tended to be stable.

Agitation is necessary for facilitating the contact of water with hydrophilic groups of small solid particles when mixing with the slurry of pecan kernel. This step results in the aggregation of all compounds which are hydrophilic so that all oils are discharged and the observation of free oils or a continuous oil phase is achieved.

Effect of Sieve Mesh on Oil Recovery

The effect of sieve mesh on OR is shown in Fig. 3. The mesh number of the sieve has a significant effect on the OR. Among all the sieve meshes studied, the OR was the highest under the condition of using a 100mesh sieve.

When the solid particles were ground to be too fine, their surface areas are increased. This may increase their capacity of holding oils so that OR can be significantly reduced. The grinder capable of performing seamless (or gapless) grinding such as wheels (or a wheel) or rollers (or a roller) combined with a sieve may give better results as compared to other type of grinders.

Extraction Procedures Established by Single Factor Experiments

Through the single factor experimentation, it was found that optimal operating procedures for recovering pecan kernel oil were as the follows. The pecan kernels were baked at 95 for 8 minutes. They were then ground to pass through a 100-mesh sieve. The accurately weighed slurry (10.00 g) and water (1.40 ml) were mixed in a 20 ml centrifuge tube. The mixture was agitated for 30 minutes at room temperature. The rest procedures starting from centrifugation were the same as that described in "2.2.1".

The optimal operating conditions of the NAM developed for the efficient recovery of pecan kernel oils were significantly different as compared with that for efficiently extracting peanuts or walnuts or sunflower seeds (Tu et al., 2017), rapeseeds (Lv and Wu, 2019a), *Camellia oleifera* seeds (Lv and Wu, 2019b), white sesame (Lv and Wu, 2020), almonds (Fu and Wu, 2019)), and soybeans (Tu and Wu, 2019) oils.

Further Optimization by Response Surface Experimentation

On the basis of single-factor experimentation previously carried out, Box-Behnken's central combination experiments were designed to investigate into multiple factor cross-effects on OR and carry out further optimization of conditions of operation. Four factors with three levels were selected in the response surface. The results showed that the OR of pecan kernels oil was significantly affected by four factors, i.e. the quantity of added water, sieve mesh number, time of agitation and temperature of baking. Table 2 indicates the results of experimentation. Table 3 shows the test result of significance of regression equation for predicting OR. Fig. 4-6 show the response surface analysis results obtained by employing Design Expert 8.0.6 software. Significant cross-effect of baking temperature and amount of added water as well as that of sieve mesh number and amount of added water on OR were found. These three factors significantly affected OR, respectively.

The final regression model in terms of coded factors established by employing SAS 8.0 in performing regression fitting on the various factors and response values is as follows:

 $\begin{array}{l} {\rm OR} = 97.33 + 1.82 \mbox{ * A} - 0.76 \mbox{ * B} + 2.74 \mbox{ * C} + 0.36 \mbox{ * D} - 1.15 \mbox{ * AB} - 0.4 \mbox{ * AC} + 0.07 \mbox{ * AD} + 1.08 \mbox{ * BC} - 0.43 \mbox{ * BD} - 0.028 \mbox{ * CD} - 2.61 \mbox{ * A}^2 - 2.15 \mbox{ * B}^2 - 3.72 \mbox{ * C}^2 - 1.09 \mbox{ * D}^2 \end{array}$

In above formula, A means baking temperature, B means quantity of added water, C means sieve mesh number, D represents agitation time.

The optimal combination of conditions of operation (experimentation) previously developed by single factor experimentation still had the highest OR (Table 2). Other condition combinations of operation (experimentation) including A-95, B-1.4 mL, C-100, D-30 min; A-95, B-1.4 mL, C-100 meshes, D-35 min; A-95, B-1.35 mL, C-100 meshes, D-30 min; and A-95, B-1.4 mL, C-100 meshes, D-30 min had a slightly lower OR, but no statistical significance of difference was found.

OR was not improved by the response surface experimentation. Therefore, the single factor experiments established condition combination of operation which was optimum and gave the highest OR.

Comparison of the New Aqueous Method with Other Methods

Table 4 compares the performance of the NAM established, with other methods and the quality of oils obtained with that of the first class oil required by Chinese National Standard. The OR obtained by NAM was obviously better than that obtained by traditional enzyme-assisted aqueous method (EAAM) extraction. Although NAM had a significantly lower OEE (P < 0.05), it had a slightly higher OR without significant difference (P > 0.05) as compared to SE. NAM also had higher OR as compared with cold pressing (Scapinello et al., 2017). Since no national standard for pecan oil acid value (AV; mgKOH/g) or peroxide value (PV; mmol/kg) was found, it was compared to the national standard of walnut oil (GB/T 22327-2008). As can be seen from the table, the pecan oil extracted by the aqueous solution method met the national standard of walnut oil. The peroxide value and acid value of the product were better than that of first class walnut oil required by the national standard. The oils were transparent, clean and colored favorably.

The de-oiled pecan meal had 27.09% of water content when 1.40 mL water was added for extracting oil. The de-oiled meal was also colored favorably (L*=55.67, a*=7.64, b*=10.67), which should be able to be applied to food recipes or the food industry.

Although the NAM used a quite low centrifugation force, a satisfactory OR was achieved. This largely decreases the purchasing or operating cost of centrifuge machine although the improvement of OR by a further increasing in centrifugation force as comparing with 1435 g employed in this study may not be ruled out. It should be noted that other methods also need centrifugation or filtration to clarify oils.

The method of extracting oils from pecan kernels by NAM produces no waste water. Drying the de-oiled meal only discharges vapor of water to the air. Therefore, the NAM is more environmental friendly as compared to EAAM (using large quantities of water previously published in literature) and SE (releasing harmful solvent to the air).

The NAM produces the de-oiled pecan kernel meal with low water content whose drying cost is greatly reduced. This is currently not achieved when the EAAM using large amounts of water .

The NAM produces oil with high quality so that it is safe for consumption without refinement (Table 4). The quality of oil produced by the NAM is far better as comparing to the crude oil obtained by SE.

Although the established NAM had a slightly lower OER as compared to SE, crude oils obtained by SE was not edible without refinement. A significant amount of neutral oil can be lost during refining process (Stalker and Wilson, 2015). Therefore, the NAM had significant higher OR as compared with SE.

Conclusion

The NAM employing a 1.4:10.0 water-to-pecan kernel slurry ratio recovered 97.73% of oils from the pecan seed kernels which had 70.47% crude oil content and produced de-oiled pecan meal with only 5.14% of oil content. The NAM did not produce waste water during oil extraction. This method had a higher OR as compared to SE and EAAM employing large amounts of water in terms of producing First Class edible oils. The high quality de-oiled pecan meal produced can be used for edible purposes without further refinement.

References

Al Juhaimi, F., Ozcan, M. M., Ghafoor, K., Babiker, E. E., & Hussain, S. (2018) Comparison of cold-pressing and soxhlet extraction systems for bioactive compounds, antioxidant properties, polyphenols, fatty acids and tocopherols in eight nut oils. *Journal of Food Science and Technology*, **55** (8): 3163-3173.

Asgar, M. A., Fazilah, A., Huda, N., Bhat, R., & Karim, A. A. (2010) Nonmeat Protein Alternatives as Meat Extenders and Meat Analogs. *Comprehensive Reviews in Food Science and Food Safety*, **9** : 513-529.

Chinese National Standard (GB/T 14488.1-2008). Determination of crude plant oil content, jointly published by General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China and Standardization Administration of the People's Republic of China.

Chinese National Standard (GB/T 5530-2005). Determination of acid value and acidity of animal and plant fats (or oils), jointly published by General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China and Standardization Administration of the People's Republic of China.

Chinese National Standard (GB/T 5538-2005). Determination of peroxide value of animal and plant fat (or oils), jointly published by General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China and Standardization Administration of the People's Republic of China.

Chinese National Standard (GB/T 22327-2008). Walnut oil, jointly published by General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China and Standardization Administration of the People's Republic of China.

Cockerham, S., Gorman, W., Maness, N., & Lillywhite, J. (2012) Feasibility assessment of investing in a pecan oil and flour processing facility using new extraction technology. Available at: http://aces.nmsu.edu/pubs/research/economics/RR778.pdf (Accessed 19-06-2018).

Environmental Protection Agency. (1999) Integrated Risk Information System (IRIS) on n-Hexane. Washington DC: National Center for Environmental Assessment, Office of Research and Development.

Fu, S., & Wu, W. (2019) Optimization of conditions for producing high quality oil and deoiled meal from almond seeds by water. *Journal of Food Processing and Preservation*, **43** (8): e14050.

Hanmoungjai, P., Pyle, L., & Niranjan, K. (2000) Extraction of rice bran oil using aqueous media. *Journal of Chemical Technology & Biotechnology*, **75** (5): 348-352.

Jin, X. F., Sun, Y. X., Zhu, L. T., & Wang, W. D. (2012) Optimization of the extraction process of alfalfa oil by enzymatic method. *Journal of Agricultural Machinery*, **18** : 42-45.

Johnson, L. A. (2000) Recovery of Fats and Oils from Plant and Animal Sources. In P. J. Wan, W. Farr (Eds.), *Introduction to Fats and Oils* (pp. 108–135). Champaign, IL: AOCS Press.

Lamsal, B. P., Murphy, P. A., & Johnson, L. A. (2006) Flaking and Extrusion as Mechanical Treatments for Enzyme-Assisted Aqueous Extraction of Oil from Soybeans. *Journal of American Oil Chemist' Society*, 83 (11): 973–979.

Latif, S., & Anwar, F. (2009) Effect of aqueous enzymatic processes on sunflower oil quality. *Journal of American Oil Chemist' Society*, 86 : 393-400.

Li, P., Gasmalla, M. A. A., Liu, J., Zhang, W., Yang, R., & Aboagarib, E. A. A. (2016) Characterization and demusification of cream emulsion from aqueous extraction of peanut. *Journal of Food Engineering*,185

: 62-71.

Liu, L., Yu, X., Zhao, Z., Xu, L., & Zhang, R. (2017) Efficient salt-aided aqueous extraction of bitter almond oil. *Journal of the Science of Food and Agriculture*, **97** (issue 11): 3814-3821.

Lv, H. J., & Liu B. G. (2005) The Utilization of Carya cathayensis Sarg Resource in the Western of Zhejiang and Southern of Anhui Province. *Journal of Ecological Economy (in Chinese)*, **05** : 97-101.

Lv, M., & Wu, W. (2019a) An advanced aqueous method of extracting rapeseed oil with high quality. *Journal of Food Process Engineering*, **42** (2): e12957.

Lv, M., & Wu, W. (2019b) Development of a new aqueous procedure for efficiently extracting high quality Camellia oleifera oil. *Industrial Crops & Products*, **138** : 111583.

Lv, M., & Wu, W. (2020) Optimization of an improved aqueous method for production of high quality white sesame oil and de-oiled meal. *Grasas y Aceites*, **71** (2): e349.

Marchetti, L., Romero, L., Andres, S. C., & Califano, A. N. (2017) Characterization of pecan nut expeller cake and effect of storage on its microbiological and oxidative quality. *Grasas y Aceites*, **68** (4): e226.

Pare, A., Nema, A., Singh, V. K., & Mandhyan, B. L. (2014) Combined effect of ohmic heating and enzyme assisted aqueous extraction process on soy oil recovery. *Journal of Food Science and Technology*,**51** (8): 1606–1611.

Qian, H. J., Yan, H. Y., Mu, H. L., Chen, H. J., & Fang, X. J. (2017) Study on the extraction technology of walnut oil by aqueous enzymatic method. *Nuclear Agricultural Journal*, **31** (07): 1365-1373.

Ravber, M., Knez, Ž., & Škerget, M. (2015) Simultaneous extraction of oil-and water-soluble phase from sunflower seeds with subcritical water. *Food Chemistry*, **166** : 316-323.

Scapinello, J., Magro, J. D., Block, J. M., Luccio, M. D., Tres, M. V., & Oliveira, J. V. (2017) Fatty acid profile of pecan nut oils obtained from pressurized n-butane and cold pressing compared with commercial oils. *Journal of Food Science and Technology*, **54** (10): 3366–3369.

Sharma, A., & Gupta, M. N. (2004) Oil extraction from almond, apricot and rice bran by three-phase partitioning after ultrasonication. *European Journal of Lipid Science and Technology*, **106** : 183–186.

Sharma, A., & Gupta, M. N. (2006) Ultrasonic pre-irradiation effect upon aqueous enzymatic oil extraction from almond and apricot seeds. *Ultrasonics Sonochemistry*, **13** : 529-534.

Stalker, T., & Wilson, R. F. (2015) Peanuts: Genetics, processing and utilization. Amsterdam: Elsevier. ISBN 1630670391

Tu, J., & Wu, W. (2019) Establishment of an aqueous method of extracting soy oils assisted by adding free oil. Grasas Y Aceites, **70** (3): e313.

Tu, J., Wu, W., Yang, J., Li, J., & Ma, X. (2017) A method of producing edible oils with high quality by water. *Journal of Food Processing and Preservation*, **41** (6): e13280.

Wang, J. P., Li, Y. N., & Ma, J. W. (1998) Study on the main nutrients in pecan kernels. *Journal of Food Science (in Chinese)*,04: 44-46.

Wang, W. Q., Bao, Y. H., Cai, Q. H., & Yu, Y. Y. (2012) Study on Extracting Technics of Walnut Oil Assisted by Ultrasonic Wave. *Journal of the Chinese Cereals and Oils Association*, **27** (12): 47-53.

Zhang, S. B., Lu, Q. Y., Yang, H., Li, Y., & Wang, S., 2011. Aqueous enzymatic extraction of oil and protein hydrolysates from roasted peanut seeds. *Journal of American Oil Chemists' Society*, **88** : 727–732.

Table 1 Summary of extraction conditions optimized by single factor experiments ^a

Baking time $(\min)^{b}$	Water added (ml/10 g) ^c	Baking temperature $()^d$	Agitation time $(\min)^e$	Sieve $\operatorname{mesh}^{\mathrm{f}}$	Brine ad
0.0	0.00	90	0	80	0.00
2.0	0.50	95	20	100	0.50
4.0	1.00	100	30	150	1.00
6.0	1.10	105	40	200	1.10
7.0	1.20	110	50	250	1.20
8.0	1.30	120	60	300	1.30
9.0	1.35	-	-	-	1.40
10.0	1.40	-	-	-	1.50
-	1.45	-	-	-	1.60
-	1.50	-	-	-	1.70
-	1.60	-	-	-	1.80
-	-	-	-	-	1.90
-	-	-	-	-	2.00

^aFor all experiments, .

^bOther parameters: baked at 110 °C, the slurry passed through a sieve having 100 meshes (154 μ m pore size), addition of 1.40 ml H₂O and agitation at room temperature (24 °C) for 30 min.

^cOther parameters: baked at 95 °C for 8 min, the slurry passed through a sieve having 100 meshes (154 μ m pore size) and agitation at room temperature for 30 min.

^dOther parameters: baked for 8 min, the slurry passed through a sieve having 100 meshes (154 μ m pore size), addition of 1.40 ml H₂O and agitation at room temperature for 30 min.

^eOther parameters: baked at 95 °C for 8 min, the slurry passed through a sieve having 100 meshes (154 μ m pore size), addition of 1.40 ml H₂O and agitation at room temperature.

 $^{\rm f}{\rm Other}$ parameters: baked at 95 °C for 8 min, addition of 1.40 ml H₂O and agitation at room temperature for 30 min.

^gOther parameters: baked at 95 °C for 8 min, the slurry passed through a sieve having 100 meshes (154 μ m pore size), brine concentration being 2.00% (w/w) and agitation at room temperature for 30 min.

^hOther parameters: baked at 95 °C for 8 min, the slurry passed through a sieve having 100 meshes (154 μ m pore size), addition of 1.40 ml brine and agitation at room temperature for 30 min.

ⁱOther parameters: baked at 95 °C for 8 min, the slurry passed through a sieve having 100 meshes (154 μ m pore size), the addition of 1.40 ml brine (2.00%, w/w) and agitation for 30 min.

Table 2 Design and results of response surface experimentation

Run	Factor 1 A:Baking temperature ()	Factor2B:Amount of water added(mL)	Factor3C:Sieve Mesh number	Factor
1	95.00	1.40	100.00	30.00
2	90.00	1.35	100.00	30.00
3	95.00	1.45	80.00	30.00
4	95.00	1.45	120.00	30.00
5	95.00	1.40	100.00	30.00
6	95.00	1.40	100.00	30.00
7	95.00	1.40	80.00	35.00
8	95.00	1.40	100.00	30.00
9	100.00	1.40	100.00	35.00
10	100.00	1.45	100.00	30.00

Run	Factor 1 A:Baking temperature ()	Factor2B:Amount of water $added(mL)$	Factor3C:Sieve Mesh number	Factor
11	100.00	1.40	80.00	30.00
12	95.00	1.40	120.00	25.00
13	95.00	1.35	100.00	25.00
14	90.00	1.40	120.00	30.00
15	90.00	1.40	80.00	30.00
16	100.00	1.35	100.00	30.00
17	95.00	1.35	80.00	30.00
18	90.00	1.40	100.00	35.00
19	95.00	1.35	120.00	30.00
20	95.00	1.40	80.00	25.00
21	95.00	1.45	100.00	25.00
22	90.00	1.45	100.00	30.00
23	100.00	1.40	100.00	25.00
24	100.00	1.40	120.00	30.00
25	95.00	1.45	100.00	35.00
26	95.00	1.40	120.00	35.00
27	95.00	1.35	100.00	35.00
28	95.00	1.40	100.00	30.00
29	90.00	1.40	100.00	25.00

Table 3 ANOVA for Response Surface Quadratic model and the analysis of variance table [Partial sum ofsquares - Type III];response: oils recovery

	Sum of	Sum of		Mean	Mean	Mean	F
Source	Source	Squares	$\mathbf{d}\mathbf{f}$	df	Square	Square	Value
Model	Model	272.43	14	14	19.46	19.46	21.52
A-Baking temperature	A-Baking temperature	39.57	1	1	39.57	39.57	43.75
B-Amount of water added	B-Amount of water added	7.01	1	1	7.01	7.01	7.75
C-Sieve Mesh number	C-Sieve Mesh number	89.82	1	1	89.82	89.82	99.31
D-Agitation time	D-Agitation time	1.55	1	1	1.55	1.55	1.71
AB	AB	5.31	1	1	5.31	5.31	5.87
AC	AC	0.64	1	1	0.64	0.64	0.71
AD	AD	0.020	1	1	0.020	0.020	0.022
BC	BC	4.67	1	1	4.67	4.67	5.16
BD	BD	0.74	1	1	0.74	0.74	0.82
CD	CD	3.025E-003	1	1	3.025E-003	3.025E-003	3.345E-003
A^2	A^2	44.13	1	1	44.13	44.13	48.80
B^2	B^2	30.08	1	1	30.08	30.08	33.26
C^2	C^2	89.57	1	1	89.57	89.57	99.04
D^2	D^2	7.72	1	1	7.72	7.72	8.54
Residual	Residual	12.66	14	14	0.90	0.90	
Lack of Fit	Lack of Fit	11.53	10	10	1.15	1.15	4.09
Pure Error	Pure Error	1.13	4	4	0.28	0.28	
Cor Total	Cor Total	285.09	28	28			

Table 4 Production efficiency of pecan oils by the advanced aqueous method and other methods as well as the characteristics of the oils produced as compared with Chinese National Standard (CNS; GB/T 22327-

Items	CNS	NAM	$\rm EAAM^{d}$	SE
OEE (%)		97.73	81.32	99.8
Smell, taste ^b	b	b		b
Transparency	C, T^{c}	C, T^{c}		C, T^{c}
AV (mg KOH/g)	[?]0.6	0.47	0.4263	0.69
PV (mmol/kg)	[?]6.0	0.34	0.2305	0.56
Residual solvent (mg/kg)	ND^{a}	ND^{a}	ND^{a}	ND^{a}
Oil content in defatted meal (%)		5.14		0.47
OR (%)		97.73	81.32	96.65

2008) for 1st class solvent extracted and refined walnut oil (Only bold items are mandatory while others are not.

^aND-undetectable; ^bHaving the inherent smell and taste of oil, no adverse smell; ^cC,T-Clarify, transparent; ^dQian et al. (2017).

Fig. 1 Process flow diagram of producing oils and defatted meal from pecan kernels by water.



Fig. 2 Effect of baking time and temperature, amount of water added and saline solution concentration on oil recovery.





Hosted file

Revised with changes tracking a new aqueous method for extracting pecan oil ja(1).docx available at https://authorea.com/users/440789/articles/541424-a-new-aqueous-method-forrecovering-high-quality-oil-and-deoiled-meal-from-pecan-kernels

Hosted file

Author's_responses_to_reviewer's_comments-JAOCS.docx available at https://authorea.com/users/ 440789/articles/541424-a-new-aqueous-method-for-recovering-high-quality-oil-and-deoiledmeal-from-pecan-kernels