Journal of Pharmacognosy and Phytochemistry
Available online at www.phytojournal.com

E-ISSN: 2278-4136
P-ISSN: 2349-8234
www.phytojournal.com
JPP 2020; 9(2): 585-590
Received: 08-01-2020
Accepted: 10-02-2020

Hetal K Bhatt
College of Food Processing Technology and Bioenergy, Anand Agricultural University, Anand, Gujarat, India

RV Prasad
College of Food Processing Technology and Bioenergy, Anand Agricultural University, Anand, Gujarat, India

Amee Ravani
College of Food Processing Technology and Bioenergy, Anand Agricultural University, Anand, Gujarat, India

Butti Prabhakar
College of Food Processing Technology and Bioenergy, Anand Agricultural University, Anand, Gujarat, India

Corresponding Author:
Hetal K Bhatt
College of Food Processing Technology and Bioenergy, Anand Agricultural University, Anand, Gujarat, India

Effect of cold milling for recovery of omega-3 fatty acid rich flaxseed oil from hydraulic press

Hetal K Bhatt, RV Prasad, Amee Ravani and Butti Prabhakar

Abstract
Flaxseed is regaining its popularity from its traditional usage as a raw material in oil production because of the reported health benefits of omega-3 fatty acids. The aim of the present investigation was to develop cold milling extraction process to get omega-3 fatty acid rich oil form flaxseed. The experiments for optimization of flaxseed oil extraction for obtaining omega-3 fatty acids rich oil were conducted as per the experimental design CCD by using hydraulic press. The effect of pretreatments feed moisture (4, 6 and 14 %), microwave power (450, 650 and 900 W) and microwave time (2, 6, 10 min). The most effective parameters for extraction of oil from hydraulic press were moisture 14 %, microwave power 600 W and microwave time 10 min where in the oil yield was 20.10 %, omega-3 fatty acid was 52.67 % and oil temperature was 29.35 %.

Keywords: Cold milling, extraction, screw press, hydraulic press

Introduction
Flaxseed is the seed from the flax plant (*Linum usitatissimum* L.), which is a member of the *Linaceae* family. The plant is native to West Asia and the Mediterranean. Flaxseed or linseed (*Linum usitatissimmum*), popularly known as Alsi, Jawas, Aksebija in Indian languages, is a blue flowering rabi crop. Flaxseed is rich in fat, protein and dietary fiber. Flaxseed contains average 30–40% fat, 20–25% protein, 20–28% total dietary fiber, 4–8% moisture and 3–4% ash, contains vitamins A, B, D and E, minerals and amino acids. The composition of flaxseed can vary with genetics, growing environment, seed processing and methods of analysis. Generally, the protein content of the seed decrease as the oil content increases.
The interest in use of the oil is due to its high content of α-linolenic acid (ALA), belonging to the ω-3 family. Especially low n-6/n-3 ratio makes it attractive for functional food and nutraceutical applications. Flaxseed oil have ω-6:ω-3 fatty acid ratio of approximately 0.3:1 (Pellizzon *et al*., 2007) [7]. Although flaxseed oil is naturally high in antioxidants such as tocopherols and beta-carotene, traditional flaxseed oil gets easily oxidized after being extracted and purified. Cold-pressed oils refer to oils that are extracted by cold-pressing plant seed with a screw press or hydraulic press. Cold-pressing is used to extract oil from plant seed instead of conventional solvent extraction method because cold-pressing does not require the use of organic solvents (CAC, 2001). Flaxseed oil is generally screw pressed and no refining is done except for sedimentation and filtration. Cold-pressing is able to retain bioactive compounds such as essential fatty acids, phenolics, flavonoids and tocopherol in the oils. Cold-pressed oils are considered as healthy oils that are important to human nutrition due to their favorable polyunsaturated fatty acid content, notably α-linolenic acid (C18:3; n-3) and linoleic acid (C18:2; n-2) (Simopoulos *et al*., 2000) [9].

Materials and Methods
Cleaned flaxseeds were pretreated before oil extraction. Moisture conditioning was done by increasing moisture content. The process as suggested by Zheng *et al*., (2003) [11] was followed with some modifications. Distilled water was sprinkled on the flaxseeds, which were thoroughly mixed by hand. The seed clumps formed by direct water addition were removed using a (9.5-mm) sieve. To decrease moisture content, flaxseeds were spread on a plate to a depth of less than 1.5 cm and then kept in a hot air oven at less than 50°C until the desired moisture content was attained.
The conditioned flaxseed was stored in a closed polyethylene bag at 5 °C for more than 5 d for equilibration.
Hydraulic press (Fig.2.) (Chatan Agro Industries, Rajkot) were used for oil extraction and the parameters used were as under. To expel the oil from flaxseed by using hydraulic press, microwave pretreatment was done to recover maximum flaxseed oil. Pretreated seeds were

used for oil extraction. Then the oil was filtered with a filter cloth and packed in amber color bottles. Oil was measured for oil yield, oil temperature and omega-3 fatty acid. Central composite design was employed to evaluate the combined effects of different parameters such as feed moisture, microwave power and microwave time.

A manual laboratory hydraulic press was used for oil extraction purposes. The extraction was done for 20 min with the pressure input exerted between 20-27 MPa. The oilseed is enclosed by a strong perforated steel cage that can apply much more pressure than an open press. Removing oil from the interior of the grain is attained by the pressure applied by a piston placed close to the cage and hydraulically operated. Oil flows through channels that increase in size from inside to outside the cage, thus avoiding any clogging with solid particles. In hydraulic pressing three stages are shown in Fig 1. (Mrema & McNulty, 1985 and Owolarafe *et al*., 2008) [5, 6]. Initially the loading stage happens before the oil begins to leave the mass of grain (oil point).

The application of compressive load causes the seeds to force the air out of the macro pores. This process continues until a critical point that occurs when the seeds respond to pressure through their points of contact. This causes the change in volume and starts the output of oil (initial stage). When the first drop of oil leaks out of the mass, it begins the second stage. (Dynamic stage), where the air is displaced by the liquid and an air/fluid mixture is extracted. The oil flow increases rapidly to its maximum, which is when the second stage ends. The last stage (final stage) begins when the maximum instantaneous flow rate, i.e. the volume is completely filled with fluid, is reached.

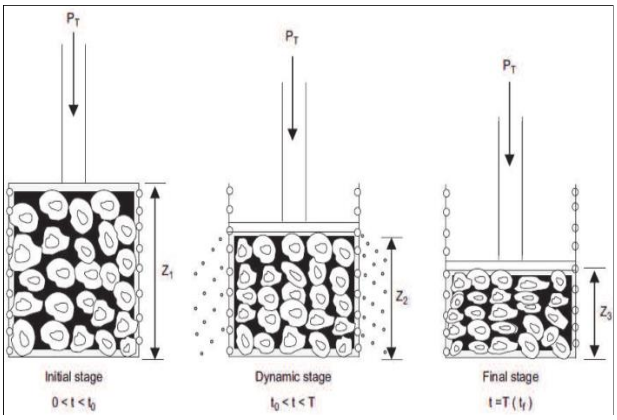


Fig 1: Stages of hydraulic expressions

Fig 2: Hydraulic press

Table 1: Variables and responses for optimization of omega-3 fatty acid rich oil from hydraulic press

Variables				Responses		
Run	**Feed moisture (%)**	**Microwave Power (W)**	**Time (min)**	**Oil yield (%)**	**Omega-3 Fatty acid (%)**	**Oil Temperature (°C)**
1	4	450	2	18.2	52.1	32.3
2	14	450	2	14.4	53.12	28.7
3	4	900	2	23.33	50.33	34.7
4	14	900	2	16.30	50.73	32.7
5	4	450	10	23.07	52.67	33.7
6	14	450	10	17.20	52.99	28.7
7	4	900	10	26.80	49.84	35.9
8	14	900	10	21.27	50.01	31.0
9	4	650	6	24.80	52.17	36.7
10	14	650	6	18.73	52.16	29.3
11	9	450	6	15.13	52.98	32.7
12	9	900	6	22.73	50.05	33.7
13	9	650	2	18.67	52.54	32.3
14	9	650	10	22.60	52.56	33.0
15	9	650	6	20.93	52.15	34.7
16	9	650	6	21.37	52.13	33.3
17	9	650	6	20.77	52.17	33.0
18	9	650	6	21.20	52.38	34.7
19	9	650	6	21.50	52.13	33.0
20	9	650	6	21.35	52.19	33.23

Flaxseed oil extraction through hydraulic press

The experiments for optimization of oil extraction pretreatments were conducted as per the experimental design and responses were collected. Oil was extracted using hydraulic press. Hydraulic press had provision for different perforated plates which facilitates developing pressure within the seed. The pressure was applied for 20 min at 27 MPa for every treatment. Effect of independent parameters *viz*. feed moisture, microwave power and microwave time on dependent parameters *viz*. oil yield, omega-3 fatty acid and oil temperature were analyzed and discussed in subsequent sections. The data pertaining the variable and responses are presented in Table 2.

To visualize the combined effects of two factors on the response, three-dimensional surface graphs were generated for each of the fitted models as a function of two variables, while keeping third variable at the central value. The linear, quadratic and interaction models were fitted to the experimental data and statistical analysis was done for each response. To compare the effects of all the factors at a particular point, the perturbation plot was drawn. The response is plotted by changing one factor over its range while holding all the other factors constant. A steep slope or curvature in a factor shows that the response is sensitive to that factor and relatively flat line shows insensitivity to change in that particular factor.

Results and Discussions

Effect of feed moisture, microwave power and microwave time on oil yield

The effect of process variables on flaxseed oil yield is depicted in Table 1. The values of oil yield obtained during experiments ranged from 14.4 to 26.80%. The maximum oil yield (26.80%) was observed on experiment no. 7 at moisture of 4%, microwave power 900 W and microwave time 10 min. The minimum oil yield (14.4%) was observed on experiment no. 2 at moisture of flaxseed 14%, microwave power 450 W and microwave time 2 min.

Among linear effects, moisture (A), microwave power (B) and microwave time (C) showed highly significant effect on oil yield ($p < 0.01$) at 1% level. Interaction effect of moisture, microwave power and microwave time (A×B, A×C, B×C) were found to be non-significant. Quadratic effect of moisture (A^2) and microwave power (B^2) were found to be highly significant ($p < 0.01$) at 1% level (Table 1.). The quadratic effect of microwave time showed non-significant effect on oil yield.

The fit of model was expressed by the coefficient of determination R^2, which was found to be 0.91, indicating that 91 per cent of variability of the response could be explained by model.

The effects of the process variables on oil yield in terms of actual levels of variables are given in regression equation as under

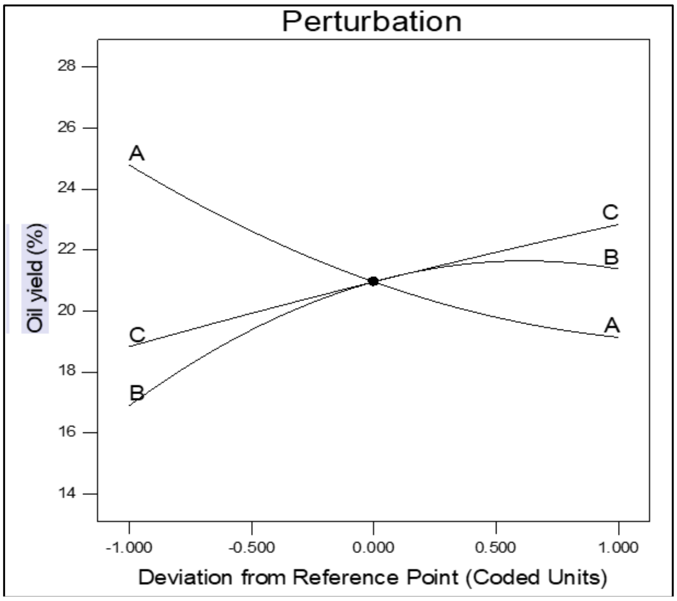
Oil yield (%) $= +20.9 - 2.82A + 2.24B + 2.0036C - 0.36AB - 0.071AC + 0.096BC + 1.01A^2 - 1.82B^2 - 0.12C^2 + 0.44ABC$

Where, A, B and C are the coded values of feed moisture (%), microwave power (W) and microwave time (min), respectively.

Effect of feed moisture on oil yield was more compared to other independent variables (Fig. 3). Decrease in oil yield was observed with increases in moisture content. There was increasing trend observed for oil yield on increasing in microwave power and microwave time. Oil yield increased with increasing in microwave power and microwave time. Yield of oil was less than that of the screw press oil extraction and this is because, temperature of flaxseed increased during oil extraction.

Fig. 4 represented effect of feed moisture and microwave power on oil yield. It was noticed that, the oil yield was lowest at 14% feed moisture. Thereafter as feed moisture decreased, the oil yield increased. Oil yield was slightly increased at highest level of microwave power. Maximum oil yield was obtained at lower level of moisture and highest microwave power.

Similar results were reported by Min and Jeong, (1995) [4] & Swern, (1982) [10]. They reported that microwave power and moisture had significant effect on oil yield. Bamgboye and Adejumo, (2007) [1] found increase in heating treatment caused protein to coagulate at a very fast rate thus reducing the viscosity significantly and adjusting the moisture content thereby leading to the release of the oil bound to them.



A=Feed moisture, B=Microwave power, C=Microwave time

Fig 3: Perturbation graph showing the effect of variables on oil yield

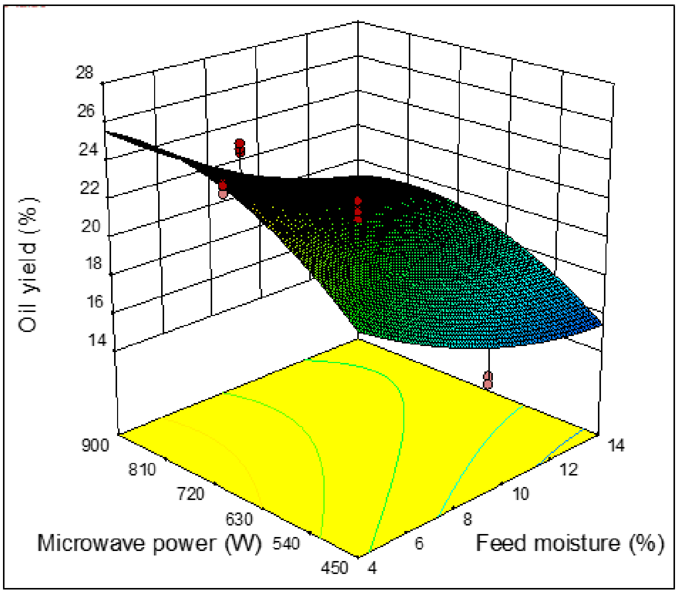


Fig 4: Effect of moisture and microwave power on oil yield

Effect of feed moisture, microwave power and time on omega-3 fatty acid

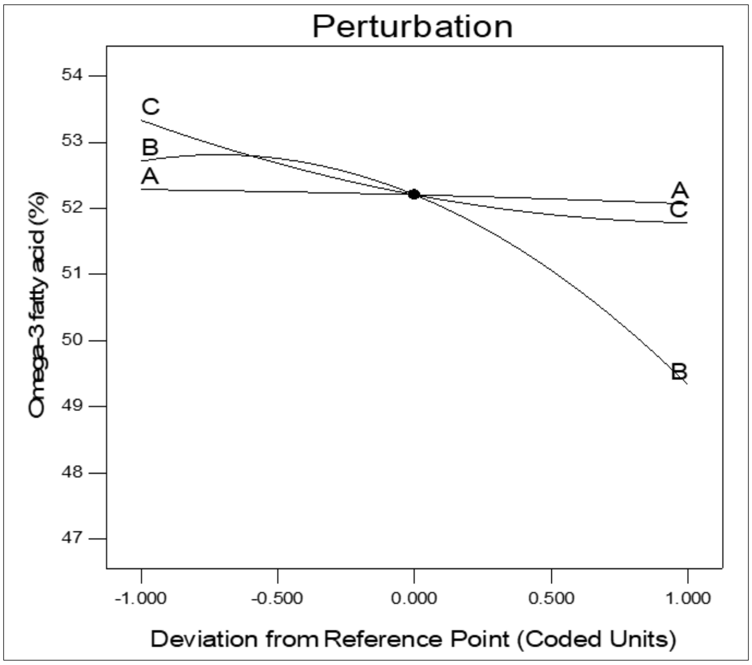
The effect of process variables on omega-3 fatty acid in flaxseed oil is depicted in Table 1. The values of omega-3 fatty acid obtained during experiments ranged from 49.84 to 53.12%.

Maximum omega-3 fatty acid (53.12%) was observed on experiment 2 at moisture of 14%, microwave power 450 W and microwave time 2 min. The minimum omega-3 fatty acid (49.84%) was observed on experiment 7at moisture of 4%, microwave power 900 W and microwave time 10 min.

Among linear effects, microwave power (B) and microwave time (C) showed highly significant effect on omega-3 fatty acid ($p < 0.01$) at 1% level. Moisture (A) showed non-significant effect on omega-3 fatty acid. Interaction effect of moisture and microwave power (A×B) was found to be non-significant. Microwave power and microwave time (B×C) showed highly significant effect ($p < 0.01$) at 1% level. Interaction effect of moisture and microwave time (A×C) was

found to be significant ($p< 0.05$) at 5% level. Quadric effect of moisture (A^2) and microwave time (C^2) were non-significant. Quadratic effect of microwave power (B^2) was found to be significant ($p< 0.01$) at 1% level of significance.
The fit of model was expressed by the coefficient of determination R^2, which was found to be 0.87, indicating that 87 per cent of variability of the response could be explained by model.
The effects of the process variables on omega-3 fatty acid in terms of actual levels of variables are given in regression equation as under

Omega-3 fatty acid (%) = $+52.20 - 0.10A - 1.69B - 0.77C - 0.22AB + 0.25AC - 0.83BC -0.029A^2 - 1.18B^2 + 0.35C^2 + 0.18ABC$
Where, A, B and C are the coded values of feed moisture (%), microwave power (W) and microwave time (min), respectively.
Perturbation graph (Fig. 5) shows the combined effect of moisture, microwave power and microwave time on omega-3 fatty acid in flaxseed oil. The microwave power had pronounced effect on omega-3 fatty acid compared to other independent variables.
Decrease in omega-3 fatty acid was observed with increase in microwave power and microwave time. There was slight increase in omega-3 fatty acid on increase of feed moisture.
Influence of microwave power and moisture on omega-3 fatty acid is shown in Figure 6. It can be noted that, when microwave power increased, omega-3 fatty acid was decreased. It can be seen that omega-3 fatty acid showed direct relation with microwave power. Maximum omega-3 fatty acid was obtained at lower level of moisture and lower microwave power. It can be seen that at increased feed moisture, omega-3 fatty acid slightly increased. There were no major changes in fatty acid profile caused by microwave treatment. These results are in agreement with those reported by Chetana *et al*., (2010) [2]. They reported that slight decreased in omega-3 fatty acid after roasting. Similar result was observed by Simbalista *et al*., (2012) [12]. They observed that there were no changes in the fatty acid profile after cold press.



A=Feed moisture, B=Microwave power, C=Microwave time

Fig 5: Perturbation graph showing the effect of variables on omega-3 fatty acid

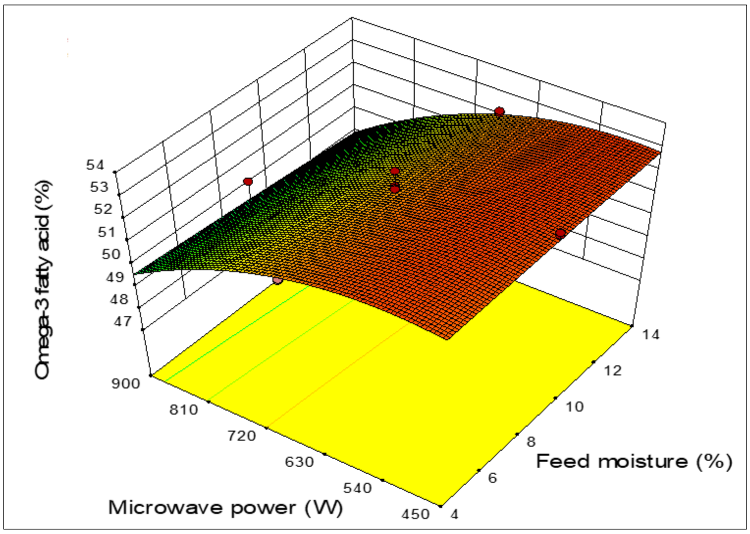


Fig 6: Effect of feed moisture and microwave power on omega-3 fatty acid

Effect of feed moisture, microwave power and microwave time on oil temperature

The effect of process variables on oil temperature is depicted in Table 1. The values of oil temperature obtained during experiments ranged from 28.7 to 36.7 (°C).

The maximum oil temperature (36.7%) was observed on experiment 9 at moisture of 4%, microwave power 650 W and microwave time 6 min. The minimum oil temperature (28.7%) was observed on experiment 2 and 6 at moisture of 14%, microwave power 450 W and microwave time 2 and 10 min, respectively.

The results showed that among linear effects, moisture (A) and microwave power (B) were highly significant ($p< 0.01$) at 1% level. Microwave time (C) showed non-significant effect. Interaction effect of moisture, microwave power and microwave time (A×B, A×C and B×C) were found to be non-significant. Quadratic effect of moisture, microwave power and microwave time (A^2, B^2 and C^2) were found to be non-significant. The fit of model was expressed by the coefficient of determination R^2, which was found to be 0.69, indicating that 69 per cent of variability of the response could be explained by model.

The effects of the process variables on oil temperature in actual levels of variables are given in terms of regression equation as under

Oil temperature (°C) = $+33.61 - 2.23A + 1.13B + 0.10C + 0.29AB - 0.45AC - 0.29BC - 0.45A^2 - 0.28B^2 - 0.78C^2$

Where, A, B and C are the coded values of feed moisture (%), microwave power (W) and microwave time (min), respectively.

Perturbation graph (Fig. 7) shows the combined effect of moisture, microwave power and microwave time on oil temperature. The feed moisture had pronounced effect on oil temperature compared to other independent variables. Increase in oil temperature was observed with decrease in moisture content.

There was slight increase in oil temperature on increase of microwave power and microwave time. Oil temperature is positively correlated with microwave power and microwave time.

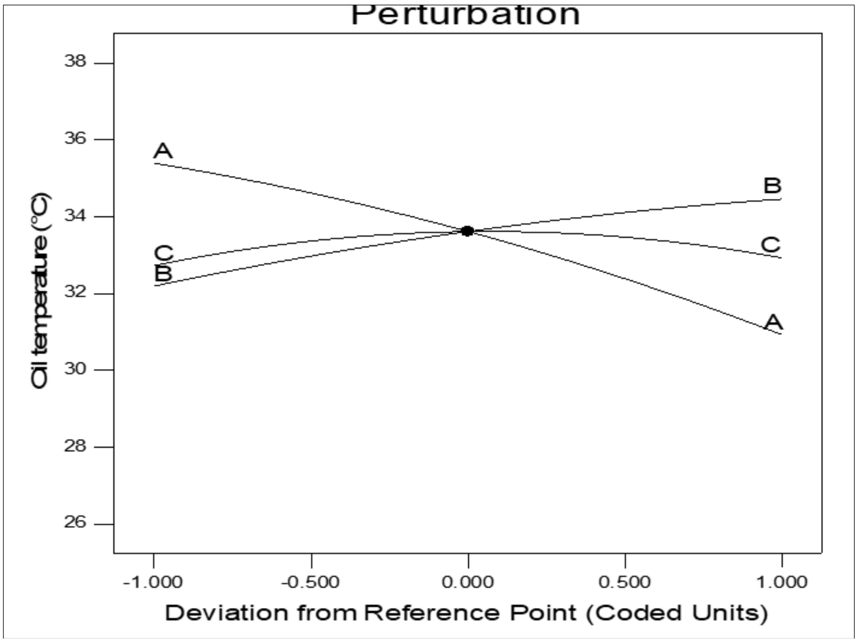


Fig 7: Perturbation graph showing the effect of variables on oil temperature

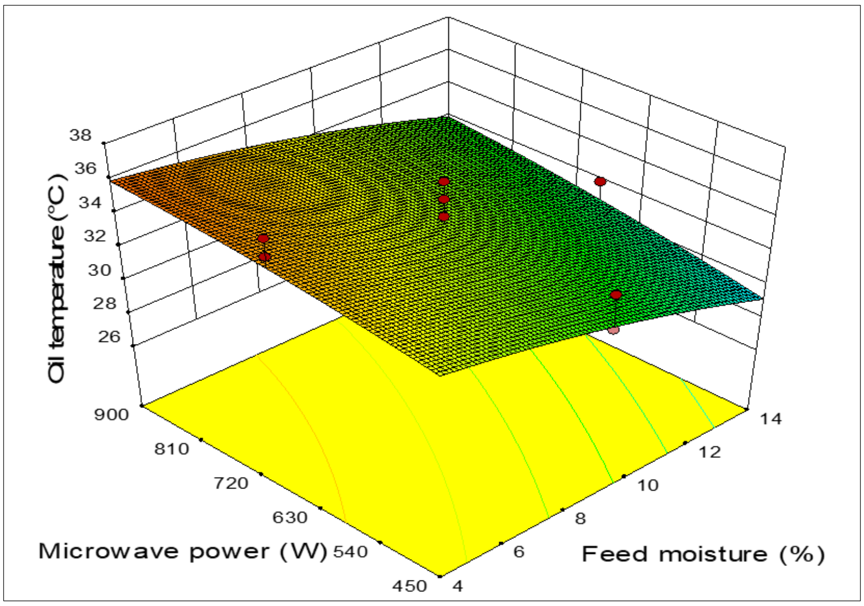


Fig 8: Effect of moisture and microwave power on oil temperature

Fig 8 represents effect of microwave power and feed moisture on oil temperature. It can be noted that, at moisture content of 4%, the oil temperature was higher.
The oil temperature increased at high microwave power but compared to screw press there was no major changes.

Optimization of hydraulic press oil extraction

Design expert software was used for simultaneous optimization of the responses. Desired goals were assigned for all the parameters for obtaining the numerical optimum values of the responses. The response parameters oil yield and omega-3 fatty acids in oil were kept maximum and oil temperature was kept minimum.

Table 2: Optimized conditions for hydraulic press oil extraction

Optimization				Optimized value	Experimental value
Constraints	**Goal**	**Lower limit**	**Upper limit**		
A-Feed moisture, %	In range	4	14	14	14
B-Microwave power, W	In range	450	900	625.81	600
C-Time, min	In range	2	10	10	10
Response					
Oil yield, %	Maximize	14	27.04	20.10	20.10
Omega-3 fatty acid, %	Maximize	49.84	53.16	52.67	52.67
Oil temperature, °C	Minimize	28.7	36.7	29.37	29.35

The co-efficient of determination (R^2) that reflects the proportion of variability in data explained and a larger R^2 values suggest a better fit of model. The R^2 values for oil yield, omega-3 fatty acid and oil temperature were 0.91, 0.87 and 0.69, respectively.
The optimum solution for extraction of oil was found with the desirability value of 0.68 having oil yield 20.10%, omega-3 fatty acid 52.67% and oil temperature 29.37% at moisture 14%, microwave power 625.81 W and time 10 min. The validation of the optimum solution was done by conducting the experiment at experimental value moisture 14%, microwave power 600 W and microwave time 10 min. Treatments for optimization with limits are shown in Table 2.

Conclusion

From the present investigation, it can be concluded that omega-3 fatty acid rich flax seed oil can be prepared from cold press system of hydraulic press. The most effective parameters for extraction of oil from hydraulic press were moisture 14 %, microwave power 600 W and microwave time 10 min where in the oil yield was 20.10 %, omega-3 fatty acid was 52.67 % and oil temperature was 29.35 %.

References

1. Bamgboye AI, Adejumo AD. Development of a sunflower oil expeller. Agricultural Engineering International: CIGR Journal. 2007; 9:1-6.
2. Chetana R, Sudha ML, Begum K, Ramasarma PR. Nutritional characteristics of linseed and its application in muffin making. Journal of Texture Studies, 2010; 41(4):563-578.
3. Joint FAO/WHO Codex Alimentarius Commission, Codex Alimentarius Commission, & Joint FAO/WHO Food Standards Programme. Codex Alimentarius: Fats, Oils and Related Products. Food & Agriculture Org, 2001, 8.
4. Min YK, Jeong HS. Optimization of batch expression of sesame oil. Journal of the Korean Society of Food and Nutrition (Korea Republic), 1995.
5. Mrema GC, McNulty PB. Mathematical model of mechanical oil expression from oilseeds. Journal of Agricultural Engineering Research. 1985; 31(4):361-370.
6. Owolarafe OK, Osunleke AS, Odejobi OA, Ajadi SO, Faborode MO. Mathematical modelling and simulation of the hydraulic expression of oil from oil palm fruit. Biosystems Engineering. 2008; 101(3):331-340.
7. Pellizzon MA, Billheimer JT, Bloedon LT, Szapary PO, Rader DJ. Flaxseed reduces plasma cholesterol levels in hypercholesterolemic mouse models. Journal of the American College of Nutrition. 2007; 26(1):66-75.
8. Jacob AA, Oluwatoyin SK, Iyabo BE, Praise O, Tertsea KM, Obozokhai O, *et al*. An appraisal of utilization level of screw press technology among Gari producers in Kwara state, Nigeria. Int. J Agric. Extension Social Dev 2019;2(2):41-46. DOI: 10.33545/26180723.2019.v2.i2a.31
9. Simopoulos AP, Leaf A, Salem Jr N. Workshop statement on the essentiality of and recommended dietary intakes for omega-6 and omega-3 fatty acids-commentary on the workshop statement. Prostaglandins Leukotrienes and Essential Fatty Acids. 2000; 63:123-124.
10. Swern D. Bailey industrial oil and fat production. 4th Edition, 1982; 2:194-200. John Wiley & Sons Inc. USA.
11. Zheng YL, Wiesenborn DP, Tostenson K, Kangas N. Screw pressing of whole and dehulled flaxseed for organic oil. Journal of the American Oil Chemists' Society. 2003; 80(10):1039-1045.
12. Simbalista RL, Frota KDMG, Soares RAM, Arêas JAG. Effect of storage and processing of Brazilian flaxseed on lipid and lignan contents. Food Science and Technology. 2012; 32(2):374-380.