Evaluación de la extracción del aceite de la Triportheus magdalenae y análisis del perfil lipídico del aceite crudo

Evaluation of oil extraction of magdalenae Triportheus and lipid profile analysis of the crude oil

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Resumen

Se evaluó el rendimiento en la extracción química del aceite de la especie íctica Arenca (Triportheus magdalenae) mediante la aplicación previa de dos tipos de digestión (ácida y acido-alcohólica), a través de un diseño experimental de bloques completamente al azar (DBCA). La extracción del aceite fue realizada por el método químico soxhlet; (adaptado del método, 960.39-AOAC para carnes) y el análisis del perfil lipídico del aceite crudo fue realizado por cromatografía Gaseosa, demostrando que los dos tipos de digestión aplicadas producen un aumento en el rendimiento de la extracción química del aceite, obteniéndose mayor rendimiento con la digestión ácida, según la Diferencia Media Significativa (DMS) con una $p<0,05$. Se determinó que la T. magdalenae, presenta una cantidad de aceite crudo en promedio de 3,23%, predominado por ácidos grasos insaturados (AGI), en un 56,77%, con proporción en ácidos grasos esenciales de la serie Omega 3 (ω-3) y omega 6 (ω-6), entre los que sobresalen el Eicosapentaenoico (EPA), el Docosapentaenoico (DPA) y el Linolénico para ω-3; el linoleíco y el araquidónico para ω-6.

Los resultados obtenidos permiten considerar la especie estudiada como viable para la obtención de aceites, de características farmacéuticas y medicinales.

Palabras claves: Arenca, Triportheus magdalenae, Aceite, Lipidico, Omega, Cromatografía.

Abstract

Performance in the chemical extraction of oil from the fish species Arenca (Triportheus magdalenae) by the prior application of two types of digestion (acid and acid - alcohol) was evaluated through an experimental design of randomized complete block (RCBD) . Oil extraction was done by soxhlet chemical method ; (adapted from the method 960.39 , AOAC meat) and the analysis of the lipid profile of the crude oil was performed by gas chromatography , showing that the two types of digestion applied produce an increase in chemical yield of the oil extraction yield better performance with acidic digestion , according to Media Significant Difference (LSD) at $P <0.05$. It was determined that the T. magdalenae , presents a number of crude oil averaged 3.23% , dominated by unsaturated fatty acids (AGI) , a 56.77 % ratio with essential fatty acids of the omega-3 (ω -3) and omega 6 (ω -6) , among which the eicosapentaenoic (EPA) , docosapentaenoic (DPA) and to ω -3 linolenic , linoleic and arachidonic to ω -6.

The results allow us to consider as a viable species studied to obtain oils, pharmaceutical and medicinal properties.

Key words: Arenca, Triportheus magdalenae, Oil, Lipid, Omega, Chromatography.

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Fish is one of the most complete foods that exists from the nutritional point of view, has significant levels of vitamins, minerals, and oils containing unsaturated fatty acids in their structure (Left Corser, P., Torres Ferrari, G., Barboza Martinez, Y., Marquez Salas, E., & Cagnasso Allara, M. 2000); also are attributed proven beneficial effects in relation to cardiovascular disease, for which international recommendations indicate increasing consumption of it (Romero, N., Robert, P., Masson, L., Luck, C., & Buschmann, L. 1996).
In Colombia, according to the International Colombia Corporation - CCI - in agreement with the Ministry of Agriculture and Rural Development - MARD (2010), the inland fisheries contributed 35% of total fish production of the country in which the basin Magdalena river reported large volumes of commercial fish landings, contributing 43.3% of the fish biomass caught in inland waters, where the Magdalena Medio region accounted for 31.8%, however, there is a high percentage of fish of low economic value that are not consumed as such, due to reasons ranging from unpleasant organoleptic characteristics to the ignorance of the nutritional properties of these fish, which the fishermen bring trapped in their networks and must be returned to the water causing job additional and unproductive, as in the case of Arenca (Triportheus magdalenae) that even today has gained commercial importance, with volumes of landings for the period 2006-2009 to 158.65 tons, according INCODER (2,010), followed still a little economically significant species.

Based on the above and taking into account current fish oil is an industrial product of high nutritional value because they contain omega-3 (Coronado, M., Vega-Leon, S., Gutiérrez, R., García fatty acids , B., & Diaz, G., 2006) and that the development of many foods is being routing to application components or properties that incorporate a healthy lipid profile in the body (Conchillo, A., Valencia, I., Puente , A., Ansorena, D., & Astiasarán, I., 2006). With the development of project performance in extracting oil from the fish species T. magdalenae evaluated and lipid profile of crude oil was analyzed, thereby seeking to propose it as a new alternative feedstock for the production of oils for industrial use.

Materials and methods

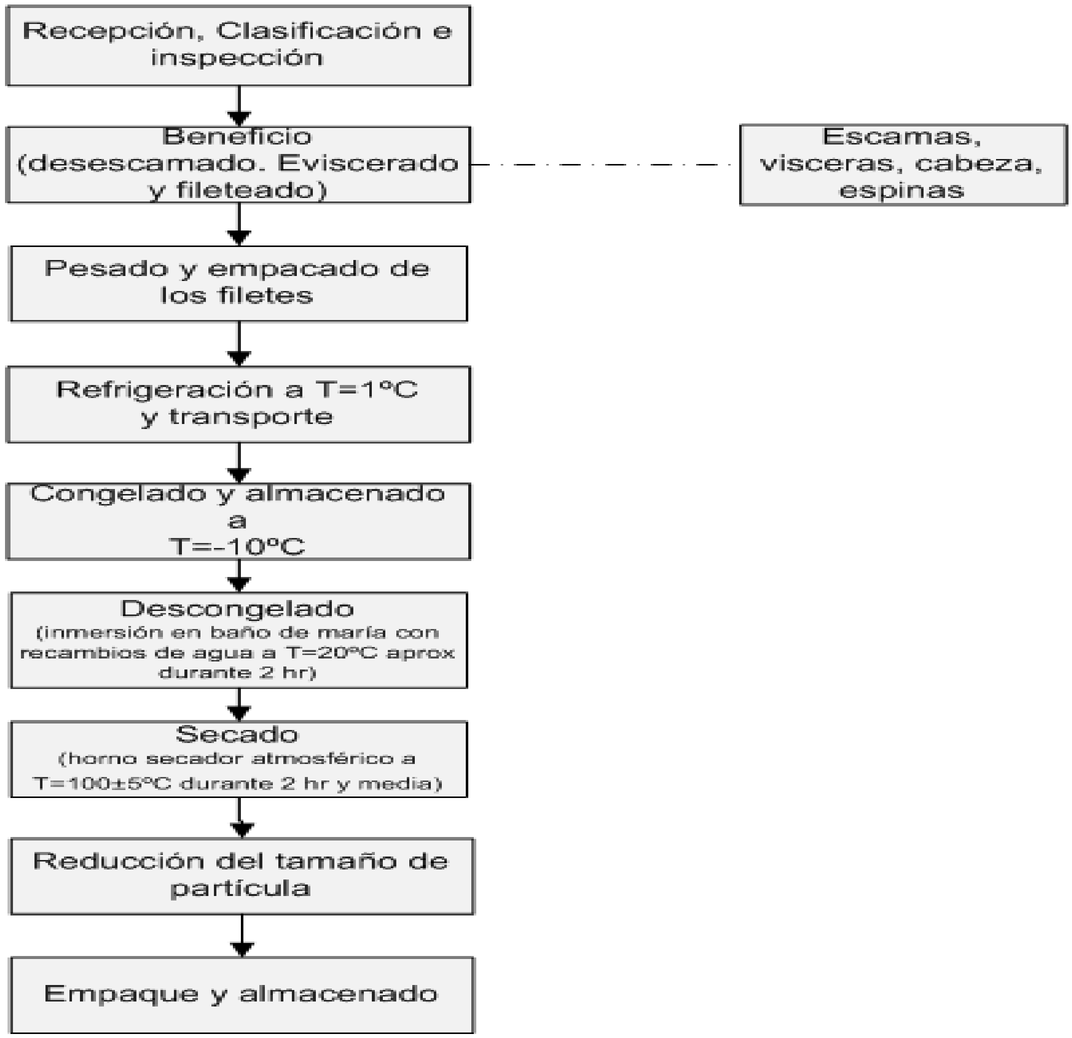
Displays

For the development of the research was carried out with the sample of the species T. magdalenae supplied by the association of fishermen, ASOPEPAL (Fishermen's Association Subdivision Palmira; NIT: 8240015002-2) Tamalameque Township - Cesar, which engaged in artisanal fishing in the Rio Magdalena and their areas of influence in this part of Colombia. For optimal sample size was considered pilot criteria (Martinez, C., 2012) to determine: industrial performance (RI), moisture, dry basis (BS) and fat (oil), in order to know the optimal amount of sample should be taken for the study, yielding an industrial yield 42.51%, 78.007 ± 0.189 moisture% which equates to a dry basis of 21,993 ± 0.189% using a moisture balance brand electronic thermal ACU, model MA-60, accuracy 0.0001 gr. Fat was determined in a range of 3.18 ± 0.05% per 100 grams of fillet approximately equivalent on a dry basis to a 14.47% oil. Based on the above, a random sample with a total weight of 4621.903 g Arenca (T. magdalenae) was used.

Processing of the species

The adequacy of the sample involved a series of stages from preparation, resulting in the production of oil as a final product, according to the methodology described by H, D, Tscheuschner (2001) as shown in Figure 1.

Figure 1 diagrams the sample flow processing



Source: H, D, Tscheuschner (2001)

Classification and inspection of the sample. The T. magdalenae was selected with a weight of 12.07 g ± 81,401 and 21,500 ± 1,654 size cm. Specimens were tested "in situ" and evaluated according to the classification table of Sensory Quality Maintaining Fish. Natural Resources Institute, (2006), as a class 5, this considering its organoleptic characteristics (color of the gills, eye color, body appearance and texture of the fish to determine its freshness)

Washing and peeling. Scales using stainless steel knives, then were washed with cold water to remove dirt (mucus from the skin of the fish) and traces of blood were removed.

Gutted. They eviscerated by complete opening of the abdominal cavity from the anus to the gills with a long cut, was later separated the back muscles and the viscera. It was observed that the species showed an advanced gonadal status (Iwaszkiw, JM, Firpo Lacoste, F., & James, A., 2010). A second washing was performed by pouring water to remove traces of viscera and walls attached to the ventral blood.

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Filleted and gutted. A longitudinal cut on both sides of the spine specimens were made, obtaining the steak and spine attached to the head. Subsequently washing fillets was performed to remove traces of blood and dirt.

Heavy and packed fillets. Fillets of the species were weighed using a balance OHAUS brand, model SP 202, capacity 200 gr gr precision 0.01 and then packed in bags of HDPE.

Cooling and transport of the fillets. To ensure the quality of all processes, the steaks were previously cooled at an average temperature of 1 ° C in expanded polystyrene boxes and then were transported to the laboratory of Chemistry and areas at the end of the Popular University of Cesar, Sectional Aguachica (UPCSA).

And stored frozen fillets. Freezing was performed by lowering the temperature of the threads and the surrounding medium, under the cryoscopic point (approx. -10 ° C) in order to maintain the frozen state for a long period of storage time.

Thaw fillets for drying. The steaks were thawed by immersion in water bath with stirring at about 20 ° C, with water exchange, measuring losses defrost (9.3015% approx.), After draining the pulp for 4 minutes.

Drying. Lipids can not be efficiently extracted with petroleum ether without prior extraction of moisture, as the solvent (petroleum ether) can not easily penetrate the tissue of the fillets wet because of this hydrophobic nature. Upon drying there is a rupture of the fat-water emulsion, causing the fat is easily dissolved in the ether oil, thus facilitating removal (Nielsen, SS 2010).

Drying the fillets of the species was made taking into account the moisture determination of pre-trials, which yielded a value of 78.007 ± 0.189% approx. Fillets were dried in a drying oven atmospheric temperature ranges 100 ° C ± 5 ° C for 2 hours until set eliminate moisture.

Particle size reduction. The effectiveness of the extraction of the oils contained in the dried samples largely depends on particle size, therefore a suitable milling is very important (Tscheuschner, HD 2001).

Dry fillets were ground in order to reduce its size, used a grinding disc, mark Victoria, to obtain a greater contact surface at the time of extracting the oil. Textured flour flowing powder was weighed for subsequent packaging is obtained.

Packaging and storage. The flour obtained was packed in sealed polyethylene bags high density capacity of 500 g, and then was stored in a glass desiccator until the time of application of oil extraction.

Evaluation of oil extraction.

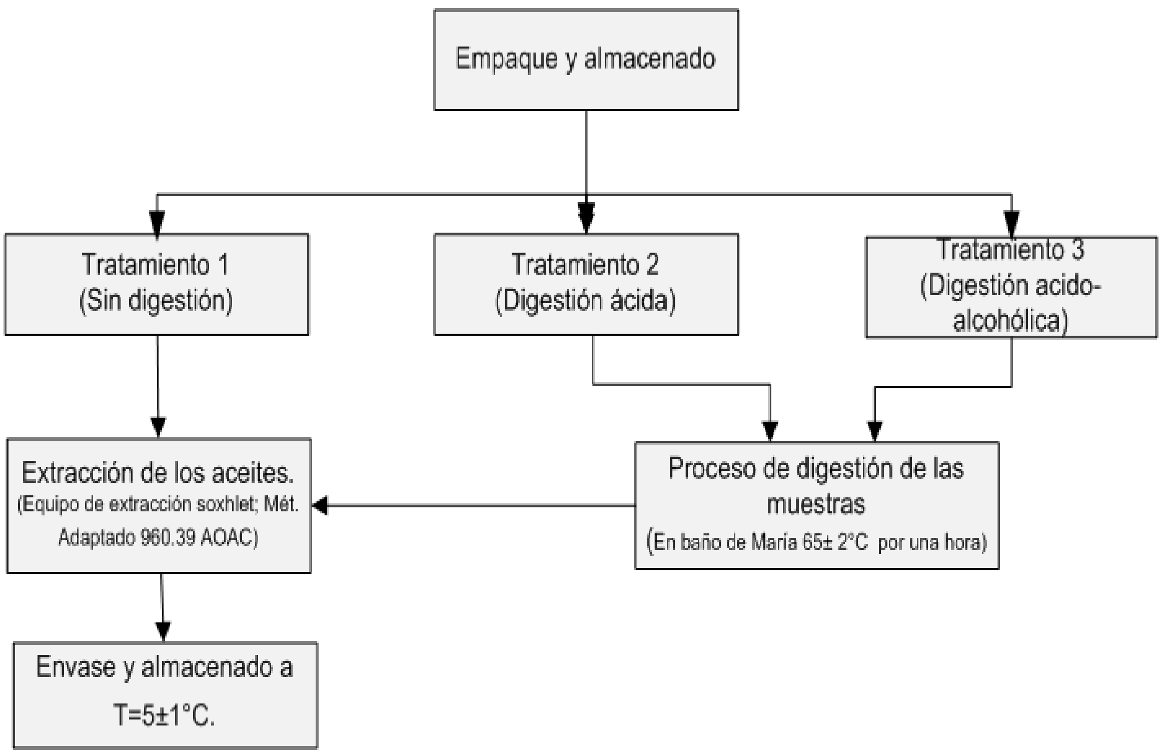
Performance in the chemical extraction of the fish species Arenca (T. magdalenae) by prior application of two types of digestion (acid and acid-alcoholic), through a block design was evaluated completely random (DBCA) (Montgomery, D. 2010), consisting of three experimental treatments applied to each of the samples. Treatment 1: no digestion (control), treatment 2: acid digestion (HCl-3N) and Treatment 3: digestion acid-alcohol (ethanol + HCl-3N) with four replicates each. extraction

the oil was conducted through the chemical method Soxhlet with petroleum ether (30-40 ° C) as solvent; (adapted from the method-AOAC 960.39, Association of Official Analytical Chemists meat).

Formulation of experimental treatments. The formulation of treatments performed considering the independent variable, ie, digestion was performed as follows: the first treatment (control) was applied without digestion, the second was applied by the addition of 20 mL of 3N hydrochloric acid, the third and final treatment, with the addition of a mixture of 20 mL of 3N hydrochloric acid + 5 mL of ethanol 95% v / v. The amounts of reactants (HCl and ethanol) used for the digestions were taken by Nielsen (2010).

Replays were obtained form four blocks of six samples, each weighing 30 ± 0.01 gr. Each block corresponds to a repetition of the experimental design as shown in Figure 2.

Figure 2. implementing flowchart experimental test



Source: The authors

For the first block simple random sampling six preweighed samples in order to obtain three samples to which is applied the design treatments in oil extraction, for examination quantitative (performance) was conducted. This was performed similarly to the other blocks. The experimental procedures are shown in Table 1, where the magnitude of survey observations for each treatment and replicate correspond to Y.

Table . Experimental setup for evaluation of oil extraction.

Tratamientos	**Bloques (repeticiones)**			
	B_1 (%)	B_2 (%)	B_3 (%)	B_4 (%)
$\mathbf{T_1}$	Y_{11}	Y_{21}	Y_{31}	Y_{41}
$\mathbf{T_2}$	Y_{12}	Y_{22}	Y_{32}	Y_{42}
$\mathbf{T_3}$	Y_{13}	Y_{23}	Y_{33}	Y_{43}
Fuente: Los autores				

Statistical analysis.

To evaluate differences between variables of each of the treatments of experimental design, statistical criteria were used as percentage analysis, analysis of variance (using less than the significance level P-value 0.05), and mean comparison test of Duncan. Supported by the statistical software SPSS version 7.5. The hypotheses were tested: (1) Null hypothesis (H0): The application of chemical digestion in oil extraction has no significant difference on the performance of T. magdalenae oil, compared with the control; (2) Alternative hypothesis (Ha): The application of chemical digestion in the extraction of oil has significant differences on the performance of T. magdalenae oil, compared with the control. The rejection of the null hypothesis involves the use of means comparison test of Duncan, in order to identify which of the experimental treatment is better in terms of oil yield.

The percentage of fat in dry weight was calculated using equation 1.

Equation 1 Percent fat in dry weight (Nielsen, 2010)

$$\% \text{ Degrasa} = (P\ A\text{-}P\ MV\ M) / (P\ Mu\ B\ S) * 100$$

Where: PMA, weight flask with oil

PMV, weight of the flask

PMuBS, sample weight dry basis

Analysis lipid profile of crude oil.

The crude oil was obtained from the extraction performed through Treatment 1 (control) applied in evaluating performance because it uses no different solvent compound needed to be considered as crude oil (Colombian Technical Standard 199, 2009) condition this is represented by a 3.23% by weight wet basis of the fillet, packed in amber test tubes and stored under refrigeration (5 ° C to 10 ° C).

Lipid Profile. Crude oil T. Magdalenae, we determined the fatty acid profile in the laboratory -SIU University Research Office - University of Antioquia. 911.35465 260 ° C: a team of Gas Chromatography (CS-MS), with brand AGILENT 7890A mass detector 5975C, HP-column was used INNOWax POLYETHYLENE GLYCO 30 mx 250 microns x 0.5 microns in which was tested in triplicate oil sample after methylation. A sample micro-liters with a concentration of approximately 20 mg / ml solution of isopropanol was injected. The results of fatty acid thrown expressed in g / 100 g of steak. (Table 5)

Results

Evaluating the chemical extraction of oil and lipid profile determined by gas chromatography was conducted.

Evaluation of oil extraction

In Table 3, the results of the extraction of the T. magdalenae each treatment are presented.

Table 3 Performance in the chemical extraction of the T. magdalenae. (Results related to% w / w dry basis)

Tratamientos	**Bloques (repeticiones)**				
	B_1 (%)	B_2 (%)	B_3 (%)	B_4 (%)	$\overline{X}$ (%)
T_1	14,36	14,55	14,63	14,35	14,47
T_2	16,45	16,05	16,25	16,20	16,24
T_3	16,00	16,25	16,20	16,35	16,20
Fuente: Los autores					

By applying the analysis of variance to the data, this revealed that the two types of digestion applied (acid and acid-alcoholic), resulting in an increased performance in the chemical extraction of the oil with respect to the control; this, because digestion previously applied to extraction, hydrolysis of generated bonds joining fat-protein and fat-other compounds, giving shapes easily removable for the solvent (petroleum ether) fat, this result is consistent studies compiled by Nielsen (2010), "The effects of digestion on the extraction of fats contained in the food," based on "the digestion of other lipids separated compounds facilitating their removal", since the increase in performance digestions oil used in this study, it was significant in comparison with the control.

Looking at the results obtained in the analysis of variance, Table 4, it can be noted that the fc >> ft (127.667 >> 5.14), indicating that there is significant difference between yields on oil treatments with a P-value <0.05, that is, that the chemical extraction of oil to the species T. magdalenae shows statistically different results in terms of performance of oil, so the best treatment was determined.

Based on the above is rejected in all cases the null hypothesis, establishing that there are significant differences between the treatments applied, whereby the use of the test for comparison of means of Duncan, with whom he established treatment became necessary two with 16.24% as the best in terms of oil yield. As shown in Figures 2 and 3.

Figure 2 categorical lines chemical treatments in oil extraction T. magdalenae species.

1ª 2ª 3ª

$$\frac{16{,}24\%}{a} \qquad \frac{16{,}20\%}{b} \qquad \frac{14{,}47\%}{c}$$

Table 4 Analysis of variance for treatments, species T. magdalenae.

Análisis de Varianza; T. Magdalenae

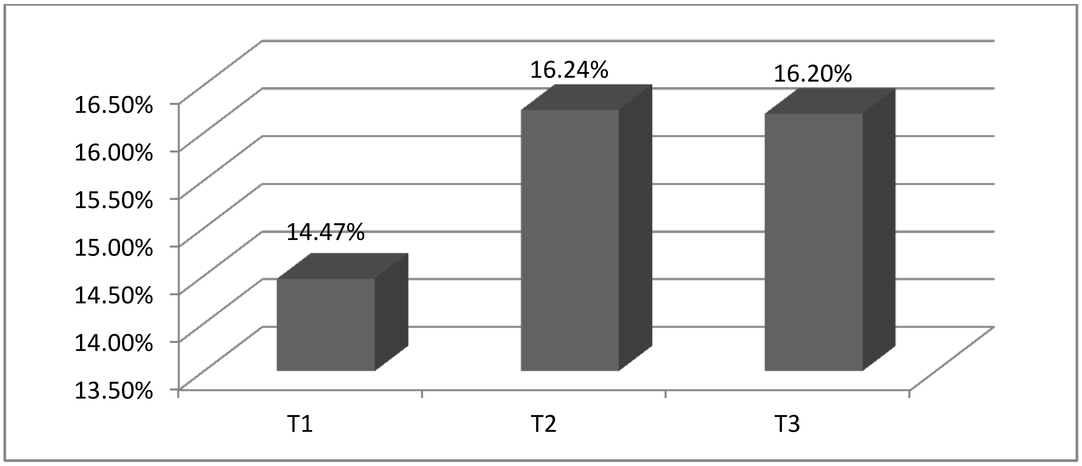
ANOVA[a]

			Método experimental				
			Suma de cuadrados	gl	Media cuadrática	F	Sig
EXTRACCI	Efectos principales	(Combinadas)	8,149	5	1,630	51,156	,0
		bloques	1,420E-02	3	4,733E-03	,149	,9
		tratamientos	8,135	2	4,067	127,667	,0
	Modelo		8,149	5	1,630	51,156	,0
	Residual		,191	6	3,186E-02		
	Total		8,340	11	,758		

a. EXTRACCI por bloques, tratamientos

Fuente: Los Autores; Software estadístico SPSS 7.5

Figure 3 Comparison of the mean for treatment in the chemical extraction of the species T. magdalenae.



Source: Authors

2.6.2 Analysis of the lipid profile of the crude oil

The results of analysis of fatty acid profile, detected in gas chromatography equipment (GC-MS), with its respective means for the kind of study, shown in Table 5.

For T. magdalenae oil, the average of the unsaturated (AGI) fatty acids represent ± 0.036 56.77%; which a 29.68% are monounsaturated and polyunsaturated to 27.09%. The unsaturated fatty acid found in a higher proportion for this species was oleic (C18: 1) 17.38 ± ω9 with 0.140% followed palmitoleic (C16: 1) with 9.58 ± 0.020%, then linolenic (C18 : 3) ω3 with 6.70 ± 0.02% and linoleic (C18: 2) 6.19 ± ω6 with 0.020%. The unsaturated fatty acid that is found in a smaller proportion was myristoleic (C14: 1) with 0.17%

Saturated fatty acids (SFA), were found in an average of 43.22 ± 0.036%. Appearing at higher concentration palmitic (C16: 0) with 16.71 ± 0.006%, followed by stearic (C18: 0) to 10.11 ± 0.030%. And tridecyl (13: 0) by 0.14% each: at lower levels lauric (C12 0) were found.

Table 5 Lipid profile determined by chromatography in the crude oil arenca (T. magdalenae)

c	
Formula abreviada	**Proporción %P/P**
C12:0	**0,14**
C13:0	**0,14**
C14:0	**3,88**
C15:0	**3,05**
C16:0	**17,3**
C17:0	**6,68**
C18:0	**10,11**
C19:0	**0,84**
C20:0	**1,09**
INSATURADOS	
C14:1	**0,17**
C16:1	**9,58**
C16::2	**0,82**
C18:1 ω-9	**17,61**
C18:2 ω-6	**6,19**
C18:3 ω-3	**6,70**
C20:1	**1,86**
C20:2	**1,17**
C20:3	**1,60**
C20:4 ω-6	**4,33**
C20:5 ω-3	**3,45**
C22:1	**0,46**
C22:5 ω-3	**2,83**
Total ácidos grasos	**43,23**

saturados	
Total ácidos grasos insaturados	**56,77**
Total ácidos grasos monoinsaturados	**29,68**
Total ácidos grasos poliinsaturados	**27,09**

Fuente: Sede de Investigación Universitaria, SIU Lab. Medellín, Antioquia

The study species, contains more polyunsaturated acids as follows; Eicosapentaenoic acid (EPA) 20: 5 ω-3, docosapentaenoic (DPA) 22: 5 ω-3 Linolenic 18: 3 ω-3, linoleic 18: 2 ω-6 and arachidonic 20: 4 ω-6, as evidenced in table 6

Table 6 fatty acids of nutritional importance of the species T. magdalenae

Ácido graso	**mg/100gr de filete**	**% ácido graso en el aceite crudo**
Mirístico	109-126	3,44-3,96
Palmítico	528-536	16,58-16,83
Esteárico	319-324	10,03-10,18
Oleico	542-564	17,03-17,73
α-Linolénico	212-215	6,65-6,75

EPA	109-111	3,42-3,47
Linoleíco	195-199	6,14-6,24
DPA	91 -92	2,82-2,84
DHA	n,d	n,d
Fuente : Los autores		

Conclusion

The effects of digestion on chemical extraction of the oil from the species of the study are positive, since the increase in yield due to applied digestions, was significant when compared to extraction performed without digestion. Therefore, to obtain a higher yield of oil is necessary to apply, prior to the extraction techniques of this type.

The results of the determination of the lipid profile, T. Magdalenae oil is considered unsaturated, which classifies it as useful raw material for the formulation and processing of food, either for human consumption or for animal consumption.

The wide variety of unsaturated fatty acids (UFA), formed in turn by AGM and AGP, especially the ω-3 and ω-6 series; detected in the oil of the study species, able to confer great nutritional value, if the many benefits it brings intake of such products, either in the diet or as a supplement to health are discussed.

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