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Extraction and characterization of oil of the pacu residue (Piaractus mesopotamicus) using ultrasonic technology

The increase in demand for fish consumption leads to the generation of a greater amount of waste, and it is necessary to seek alternatives to reuse of this organic load. In this way, the objective of this research was to evaluate the extraction of oil from the pacu residue by ultrasonic energy. The oil extraction was evaluated using a wet and dry raw material at 70 °C, from a complete factorial planning 2^2, where the variables were sample solvent ratio and time. The oils obtained in all the extraction conditions were analyzed as a function of the acidity in oleic acid (%), and the oil obtained in the best condition was submitted to the fatty acid profile. In the extraction smaller times and larger proportions of solvent sample contributed to obtain higher yields in oil. The oils obtained had low acidity and a high content of unsaturated fatty acids, with a ratio of unsaturated and saturated and ?-6/?-3 within the desired limits. In this way, ultrasound proved to be a useful tool for obtaining good quality pacu residue oil.

Keywords: Fish; Ultrasound; Acidity; Fatty Acids.

Extração e caracterização de óleo do resíduo de pacu (Piaractus mesopotamicus) usando tecnologia ultrassônica

O aumento da demanda pelo consumo de peixe leva à geração de uma quantidade maior de resíduos, sendo necessário buscar alternativas para o reaproveitamento dessa carga orgânica. Dessa forma, o objetivo desta pesquisa foi avaliar a extração de óleo do resíduo do pacu por energia ultrassônica. A extração do óleo foi avaliada utilizando uma matéria-prima úmida e seca a 70 ° C, a partir de um planejamento fatorial completo 2^2, onde as variáveis ??foram razão e tempo de solvente da amostra. Os óleos obtidos em todas as condições de extração foram analisados ??em função da acidez em ácido oleico (%), e o óleo obtido nas melhores condições foi submetido ao perfil de ácidos graxos. Na extração, tempos menores e proporções maiores de amostra de solvente contribuíram para obter maiores rendimentos em óleo. Os óleos obtidos apresentaram baixa acidez e alto teor de ácidos graxos insaturados, com uma proporção de insaturados e saturados e ?-6/?-3 dentro dos limites desejados. Dessa maneira, o ultrassom mostrou-se uma ferramenta útil para a obtenção de óleo de resíduo de pacu de boa qualidade.

Palavras-chave: Peixe; Ultrassom; Acidez; Ácidos graxos.

Topic: **Engenharia Química**

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INTRODUCTION

Fish are highlighted for being excellent sources of protein, polyunsaturated fatty acids and minerals. However, with increasing consumer demand, the amount of waste from fish processing industries grows in equal and/or greater proportion (MONTEIRO, 2013). Alongside, pacu production shows a fast-growing growth in the Brazilian aquaculture sector, in 2017, the country was responsible for more than 13.15 tons of pacu, and approximately 3.47 tons were produced by the Midwest region (BRASIL, 2017).

With this high pacu production rate, large amounts of waste are generated during processing, which are characterized by having a relatively low added value such as head, fins, skin, viscera and scales, that can be used properly, providing income for anglers or processing establishments, as well as minimizing the impact on the environment, and introducing new products to the market, since oil extracted from these wastes can be used for biodiesel production or even for human consumption (MARTINS et al., 2014).

Ultrasonic technology has been exploited by many researchers because it is considered relatively simple and inexpensive, as well as environmentally friendly, because it relates two basic requirements for obtaining the oil: shorter extraction time and reduced amount of solvent generating no waste (TSUKUI et al., 2014). The technique of extraction by ultrasound in obtaining oil stands out for reducing the process steps, exponentially maximizing the quality of the final product, since it presents improvement in mass transfer, preserving the characteristics of the food because it is an effective procedure in breakage of tissues, facilitating the removal of oil (AWAD, 2012).

However, despite the advantages and simplicity of the oil extraction process using ultrasonic energy and the extensive knowledge regarding fish residue, this technology is still little explored for this purpose, since the works reported in the literature use traditional methods for oil extraction from this residue. Therefore, the present study aimed to obtain oil from pacu (*Piaractus mesopotamicus*) residue using ultrasonic energy, evaluating the operational variables that interfere in the extraction. Furthermore, characterizing the oil quality obtained through the acidity index and its function, fatty acid profile, allowing the addition of value to an abundant, nutritionally rich waste with high environmental impact, since it has a high organic load.

MATERIALS AND METHODS

Obtaining and characterization of pacu residue

Pacu residues were purchased from anglers in Barra do Bugres/MT, in a refrigerated environment (4 °C). The residues consisted mainly of head, spine, fins, leather, scales and tail, which were powdered using an industrial blender and stored under freezing (-8 °C) until the time of analysis. To characterize the fish residue, pH, acidity, moisture and ash analyzes that were performed, it was employed the methodology of the Adolf Lutz Institute (2008). For proteins determination, the Detmann (2012) method was used.

Lipid extraction and characterization

The ultrasound lipid extraction technique was performed using petroleum ether as a solvent. The samples previously dried at 70 ° C, as suggested by Rigueto et al. (2017) were submitted to ultrasonic bath (Quimis, Q335D) with a frequency of 40 kHz. The reaction mixture formed was extracted, under different operating conditions, according to complete factorial design 2^2, with three central points, totaling 7 experiments (Table 1), where the variables studied were extraction time and mixing ratio (sample mass: volume of solvent) according to a study developed by Barbalho et al. (2013), in the extraction of pequi oil using ultrasonic energy. At the end of the extraction process, the excess solvent was evaporated in an oven at 70 °C to constant weight to quantify the mass of extracted lipids.

Table 1: Variables and levels studied in the complete factorial design 2^2, with three central points used in the extraction of pacu residue oil.

Levels	Time (h)	Mixing ratio Sample: solvent (g mL^{-1})
-1	1	1:2
0	2	1:4
1	3	1:6

The determination of the extracted oil content was made by the mass difference between the initial sample weight and the final sample weight after solvent extraction and evaporation. To express the results, the data were transformed as a percentage of the initial sample weight. The characterization of the oil was based on the acidity index according to the methodology of the Adolf Lutz Institute (2008) and measured as oleic acid (%) for each drying temperature (50, 60 and 70 °C) for extractions in ultrasonic bath. The samples were analyzed in triplicate, and the experimental design was completely randomized, and the results of the evaluations were compiled and analyzed by statistic software, version 7.0.

In addition to the fatty acid profile, the fatty acid methyl esters were analyzed in a gas chromatograph (Varian, 33800, equipped with a flame ionization detector and CP - 7420 fused silica capillary column (Select FAME) (100 m in length). 0.25 mm internal diameter and 0.25 µm cyanopropyl). The flow of H_2 (carrier gas) was 1.0 mL min^{-1} with 30 mL min^{-1} N_2 (make up) and 30 and 300 mL min^{-1} for H_2 and synthetic air for detector flame, respectively. The injected volume was 2.0 µL using split 1:80, with injector and detector temperatures of 220 and 240 °C, respectively, while the column at 165 °C for 18 min and raised to 235 °C at 4 °C min^{-1} maintained for 14.5 min. Results were expressed as fatty acid percentages (AGUIAR, 2011).

RESULTS AND DISCUSSION

The centesimal composition generally portrays the nutritional value of the analyzed sample. Table 2 shows a comparison of the physicochemical analyzes between the waste used in this work and the residues of various fish found in the literature. In the analysis of Table 2, it can be observed that the moisture content obtained in the present study (53.33%) was lower than that found for other samples of fresh fish residues. However, the ash content was higher when compared to Aguiar (2011) and lower than Souza et al.11. Franco et al.12 also obtained lower ash values for tilapia (1.82%), pacu (1.32%) and tambaqui (1.45%) skin.

Guzmán (1994) emphasizes that these differences in moisture and ash content may be related to

genetic, morphological, physiological or exogenous factors (climate and season). Also, according to Boscolo et al. (2011) the protein content presented higher value compared to the residues of pescada-olhuda and tilapia, and this variation in protein content occurs due to species and eating habits of the fish, since pacu is omnivorous and it also feeds on other fish. Oil extraction was performed using ultrasonic technology. Table 3 shows the oil yields obtained in an ultrasonic bath.

Table 2: Physicochemical characterization of pacu residue of the present study, compared with fillet residues of pescada-olhuda and tilapia viscera.

Parameters*	Residue (a)	Residue (b)	Residue (c)
Specie	Pacu	Pescada-olhuda	Tilápia
pH	7,21±0,09	-	-
Acidity (%)	41,14±3,52	-	-
Moisture (%)	53,33 ±1,37	68,6	64,4
Ashes (%)	4,44 ±0,03	7,0	1,3
Protein (%)	22,06 ±3,00	19,0 ±1	6,3 ±0,6

Values expressed as mean ± standard deviation. The standard deviation is only found in residue a because it corresponds to the values of the present work. Other residues refer to data published by other authors. (a) present work; (b) Aguiar (2011); (c) Souza et al. (2005). * Results expressed on wet basis.

Table 3: Complete factorial design matrix 2^2, with three central points for pacu residue oil extraction, with actual and coded variables and responses expressed in terms of oil yield.

Trial	Time (h)	Sample Reason: Solvent (g mL^{-1})	Oil (%)	Acidity in oleic acid (%)
1	1 (-1)	1:2 (-1)	26,09	0,60 ±0,01
2	3 (1)	1:2 (-1)	24,42	0,67 ±0,02
3	1 (-1)	1:6 (1)	31,44	0,59 ±0,01
4	3 (1)	1:6 (1)	29,16	0,63 ±0,01
5	2 (0)	1:4 (0)	29,83	0,95±0,01
6	2 (0)	1:4 (0)	31,20	0,99 ±0,02
7	2 (0)	1:4 (0)	29,75	0,97 ±0,03

The good reproducibility of the results obtained in the experimental design (Table 3) can be observed in the central points (tests 5, 6 and 7), since the obtained values are very close, showing good repetition and low experimental error, with an average of 30.26 ± 0.82% for the percentage of oil obtained and 0.97 ± 0.02% for the acidity in oleic acid.

In Table 3 it is possible to observe that the oil contents varied from 24.42 to 31.44%, and the highest oil content was extracted in the 3° trial and in the central points. However, when analyzed the extraction time, trial 3 presented a difference of 1h of extraction, compared to the trials of the central point, which was 2 h. Thus, the shorter the time the higher the oil content found. Regardless of the extraction time, the lowest oil content was obtained when the lowest sample and solvent ratio was used, showing that this is the variable that most influences the oil extraction of fish residue using ultrasonic energy, this finding is observed in Figure 1, which shows the influence of the variables studied on oil content.

Pareto diagram confirms that the effect of the sample: solvent variable exerts the greatest influence on the extraction of pacu residue oil, this influence being positive, while the influence of time is negative. Thus, the shorter the extraction time and the higher the sample and solvent ratio, the higher the percentage of oil extracted. However, this influence is not statistically significant, showing that there is no statistical relationship between the dependent (% oil) and independent (time and sample and solvent ratio) variables. This answer can also be verified by the Analysis of Variance (ANOVA) which presented a coefficient of

determination (R^2) of only 0.30, while the indicated one is at least 0.90.

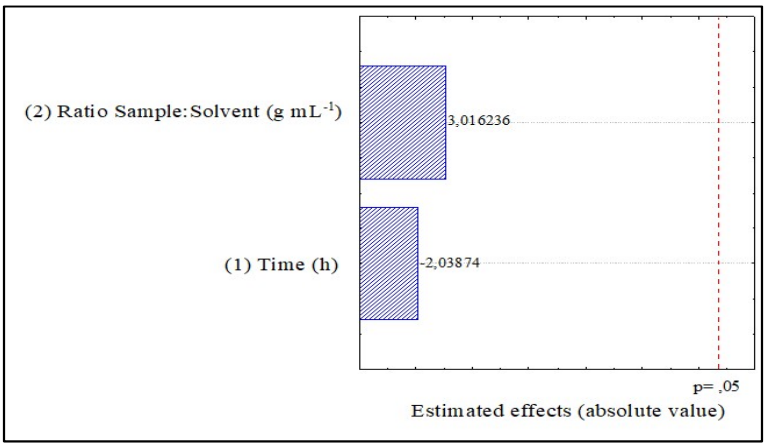

Figure 1: Pareto diagram representing the effect of sample: solvent ratio and time on oil extraction from pacu residue.

The state of conservation of the oil is closely related to the nature of the extraction, as well as the quality of the raw material, which can be assessed by the acidity index. Storage also requires specific care as triglyceride decomposition increases when exposed to heating or lighting (STEVAN JUNIOR et al., 2015). In relation to the acidity (Table 3), it can be seen that the oils obtained by extraction in an ultrasonic bath presented acidity values in oleic acid according to the maximum stipulated by RIISPOA, which is 3% (BRASIL, 1952). Besides that, Bery (2012) states that the acidity index has a significant influence on the quality and condition and conservation of the oil, being suitable acidity values lower than 1%, so that all experimental conditions evaluated in the present study made it possible to obtain a fish oil with a good physical chemical quality, regarding acidity in oleic acid.

However, the best oleic acid levels found in oils extracted by the ultrasonic bath were from trials 1 and 3, showing that the shorter the extraction time, the lower the acidity and consequently the better the oil quality. Thus, to evaluate the fatty acid profile of the oil obtained from fish waste and using ultrasonic energy, the oil obtained with the experimental conditions employed in the trial 3 (1 h and 1: 6 solvent sample ratio) was chosen, as this made it possible to obtain higher oil content and lower acidity index in oleic acid.

Ultrasound proved to be a good alternative for lipid extraction, since the acidity values obtained by this method are significantly lower when compared to the study by Rigueto et al. (2017), which obtained 18.27% oleic acid acidity of the residue of dried pacu at the same temperature (70 °C) using soxhlet as the extraction method. The fatty acid profile of the oil extracted (trial 3) with pacu residue ultrasonic energy is shown in Table 4.

According to the results presented in Table 4, 21 fatty acid types were identified in the oil extracted from pacu residue with predominance of oleic acid (31.08%), palmitic acid (20.81%), linolenic acid (11.02%) and linoleic acid (9.77%). Machado et al. (2013) report that freshwater fish have generous amounts of linoleic and linolenic acids, which may explain the fact that linoleic acid is among the predominant fatty acids in pacu waste oil. The results obtained are similar to those reported by Segura (2012) who analyzed the fatty acid profile of three different freshwater fish (pacu, curimbatá and rainbow trout) and obtained the predominance of oleic, palmitic and linoleic acid in all oils analyzed.

Table 4: Fatty acid composition of oil extracted from pacu residue by assisted extraction of ultrasonic energy

Fatty acid	% (w/w)
Lauric Acid (C12: 0)	1,52 ± 0,05
Myristic acid (C14: 0)	2,96 ± 0,02
Pentadecylic acid (C15: 0)	0,31 ± 0,01
Palmitic acid (C16:0)	20,81 ± 0,53
Margaric acid (C17:0)	5,75 ± 0,02
Stearic acid (C18:0)	7,65 ± 0,32
Arachidic acid (C20:0)	1,04 ± 0,04
Henicosanoic acid (C21:0)	0,39 ± 0,01
Lignoceric Acid (C24:0)	0,13 ± 0,00
Total Saturated Fatty Acid	**40,56**
Palmitoleic Acid (C16:1)ω-7	0,61 ± 0,02
Heptadecenoic Acid (C16:1)ω-9	0,62 ± 0,01
Oleic acid (C18:1)ω-9	31,08 ± 0,89
Vaccenic acid (C18:1)ω-7	3,10 ± 0,08
Gondoic acid (C20:1)ω-9	0,88 ± 0,07
Total Monounsaturated Fatty Acids	**36,29**
Linoleic acid (C18:2)ω-6	9,77 ± 0,07
Linolenic Acid (C18:3)ω-3	11,02 ± 0,06
Dihomo γ linolenic acid (C20:3)ω-6	0,29 ±0,01
Eicosatrienoic acid (C20:3)ω-3	0,78 ±0,01
Arachidonic acid (C20:4)ω-6	0,63 ±0,01
Eicosapentaenoic acid (C20:5)ω-3	0,29 ± 0,02
Docosahexaenoic acid (C22:6)ω-3	0,39 ± 0,02
Total Polyunsaturated Fatty Acids	**23,17**
Total Unsaturated Fatty Acids	59,46
Ω-3 total	12,48
Ω-6 total	10,69
Ω-7 total	3,71
Ω-9 total	32,58
Unsaturated / Saturated Ratio	1,46
Ratio ω-6 / ω-3	0,86

Pacu residue oil showed a total of 40.56% (saturated fatty acids) and 59.46% (unsaturated fatty acids), showing that pacu residue oil can be an alternative for the consumption of polyunsaturated fatty acids. unsaturated. In addition, a ratio of 1.46 and 0.86 was obtained for unsaturated / saturated GA and ω-6 / ω-3, respectively.

According to the Department of Health and Social Security (1984), diets that have an established/saturated GA ratio higher than 0.45 are considered nutritionally healthy. Also, according to the Department of Health & Social Care (1994) the ratio of AG ω-6/ω-3 should be less than 4, characterizing a meat with desirable lipid content for human nutrition. Therefore, the oil extracted from the pacu residue is within these specifications.

CONCLUSIONS

Faced to the proposed objectives and results obtained in the present study, it is concluded that the method of extraction in ultrasonic bath is a viable alternative for lipid extraction, since it allows obtaining oil in relatively short time and oil obtained from this technique presents low acidity index in oleic acid.

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REFERENCES

AGUIAR, A. C.. **Extração de lipídios de resíduos de filetagem de pescada-olhuda (cynoscion striatus) utilizando tecnologia supercrítica.** Dissertação (Mestrado) - Universidade Federal de Santa Catarina, Florianópolis, 2011.

AWAD, T. S.; MOHARRAM, H. A.; SHALTOUT, O. E.; ASKER, D.; YOUSSEF, M. M.. Applications of ultrasound in analysis, processing and quality control of food: A review. **Food research international**, v.48, n.2, p.410-427, 2012.

BERY, C. C.. Estudo da viabilidade do óleo de vísceras de peixes marinhos (Seriola dumerlii (arabaiana), Thunnus ssp (atum), Acomberomorus cavala (cavala) e Carcharrhinus spp (cação)) comercializados em Aracaju-SE para a produção de biodiesel. **Gestão, Inovação e Tecnologias,** v.2, n.3, p.297-306, 2012.

BOSCOLO, W. R.; SIGNOR, A.; FREITAS, J. D.; BITTENCOURT, F.; FEIDEN, A.. Nutrição de peixes nativos. **Revista Brasileira de Zootecnia**, v.40, p.145-154, 2011.

BARBALHO, T. C. S.; PINHEIRO, A. D. T.; LUCENA, I. L.. Estudo da extração do óleo de pequi (Caryocar brasilienses camb.) assistida por ultrassom. In: CONGRESSO BRASILEIRO DE QUÍMICA, 53. **Anais.** 2013.

HMSO. Department of Health & Social Care. **Nutritional aspects of cardiovascular disease**. London: HMSO, 1994.

DHSS. Department of Health and Social Security. **Diet and cardiovascular disease**. London: HMSO, 1984.

DETMANN, E.. **Métodos para análises de alimentos:** Instituto Nacional de Ciência e Tecnologia de ciência animal. Visconde do Rio Branco: Suprema, 2012.

FRANCO, M. L. R. S.; FRANCO, N. P.; GASPARINO, E.; DORADO, D. M.; PRADO, M.; VESCO, A. P. D.. Comparação das peles de tilápia do Nilo, pacu e tambaqui: Histologia, composição e resistência. **Archivos de zootecnia**, v.62, n.237, p.21-32, 2013.

GUZMÁN, E. S. C.. **Bioquímica de Pescados e Derivados**. Jaboticabal: FUNE, 1994.

IAL. Instituto Adolfo Lutz. **Métodos Físicos Químicos para Análise de Alimentos**. São Paulo: IAL, 2008.

STEVAN JUNIOR, S. L.; PAITER, L.; GALVÃO, J. R.; ROQUE, D. V.; CHAVES, E. S.. Sensor and methodology for dielectric analysis of vegetal oils submitted to thermal stress. **Sensors**, v.15, n.10, p.26457-26477, 2015.

MACHADO, B. A.; REIS, J. H. O.; NUNES, I. L.; PADILHA, F. F.; DRUZIAN, J. I.. Determinação do perfil de ácidos graxos por cromatografia gasosa, composição proximal e valor calórico de carne de avestruz (Struthio camellus). **Alimentos e Nutrição Araraquara**, v.24, n.2, p.216, 2013.

MARTINS, G. I.; SECCO, D.; TOKURA, L. K.; BARICCATTI, R. A.; DOLCI, B. D.; SANTOS, R. F.. Potential of tilapia oil and waste in biodiesel production. **Renewable and Sustainable Energy Reviews**, v.42, p.234-239, 2015.

MONTEIRO, M. M. G.. **Aproveitamento de resíduos de tilápia (Oreochromis niloticus) para elaboração de novos produtos com valor agregado.** Tese (Doutorado) - Universidade Federal Fluminense, Niterói, 2013.

RIGUETO, C. V. T.; LOSS, R. A.; FREITAS, T. S. M.; BEVILAQUA, N. C. O.; BRUM, F. B.. Influência da temperatura de secagem do resíduo de pacú (Piaractus mesopotamicus) para obtenção de óleo. **Revista Ibero-Americana de Ciências Ambientais,** v.8, n.1, p.218-225, 2017.

SEGURA, J. P.. **Extração e caracterização de óleos de resíduos de peixe de água doce.** Dissertação (Mestrado) - Universidade de São Paulo, Pirassununga, 2012.

SOUZA, N. E.; MATSUSHITA, M.; FRANCO, M. R. B.; PRADO, I. N.; VISENTAINER, J. V.. Composição química, perfil de ácidos graxos e quantificação dos ácidos a-linolênico, eicosapentaenóico e docosahexaenóico em vísceras de tilápias (Oreochromis niloticus). **Acta Scientiarum. Technology**, v.27, n.1, p.73-76, 2005.

TSUKUI, A.; REZENDE, C. M.. Extração assistida por micro-ondas e química verde. **Revista Virtual de Química**, v.6, n.6, p.1713-1725, 2014.