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OPTIMIZATION AND INNOVATION OF THE CLARIFICATION PROCESS OF VEGETABLE OILS: A REVIEW

Renan Felicissimo Marangoni[a], Tatiane Viana Dutra[b], Leila Larisa Medeiros Marques[c] and Flávia Aparecida Reitz Cardoso*[a]

[a]Post-Graduation Program of Technological Innovation (PPGIT), Federal University of Technology – Paraná (UTFPR), Campo Mourão, Paraná, 87301-005, Brazil; [b]Post-Graduation Program of Food Science, State University of Maringá (UEM), Maringá, Paraná, 87020-900, Brazil; [c]Department of Food Engineering, Federal University of Technology – Paraná (UTFPR), Campo Mourão, Paraná, 87301-005, Brazil

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****Corresponding author:***
Flávia Aparecida Reitz Cardoso

ABSTRACT

The clarification is a process of adsorption of pigments and impurities present in vegetable oils. This process occurs in a conventional way using a high-cost mineral adsorbent. Because of this adversity, opted to carry out a bibliographical review to gather information regarding the optimization of the clarification process. Sixteen studies were selected that deal with the optimization of the clarification of vegetable oils and the use of new technologies to improve process performance. The studies show that the raw material influences the process parameters, and it is dependent of factors such as temperature, reaction time and adsorbent dosage. The use of new auxiliary technologies in the clarification process are potential process optimization agents because they show satisfactory results compared to the conventional adsorption process. The literature consulted presents relevant data regarding the optimization of the process of clarification of vegetable oils.

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INTRODUCTION

The importance of innovation continues to rise in the academic, social and organizational environment, in the last, as it is a way to achieve success in the current markets or as a way of creating new markets(Nagano, Stefanovitz, & Vick, 2014). According to Bessant and Tidd,(Bessant & Tidd, 2009) innovating in processes involves improving existing processes, such as reducing waste, increasing efficiency or changing the way in which the process is operated. As reported by the Organization for Economic Co-operation and Development(OECD, 2005) in it's manual of practices and methods of innovation known as the Oslo Manual, innovating in processes brings significant changes in the method of production or distribution of a product or service. (Volberda, Van Den Bosch, & Heij (2013)report that innovation is the main agent that promotes progress and prosperity and can be applied in the most diverse fields of knowledge, such as in the development of new technologies, new knowledge, new processes and new products. In the processing of oils extracted from the most diverse vegetable sources to obtain vegetable oil destinated for human consumption, a series of chemical and physical steps constituted in the process known as refining of vegetable oils is necessary to obtain a final product free of impurities and quality assurance. In the industrial process of refining of vegetable oils, the clarification step is in charge of removing oxidation products (hydroperoxides) and pigments such as chlorophyll, which, in addition to promoting depreciation of the final product when in high concentration, is an oxidizing agent of fat when exposed to ultraviolet radiation, which seriously impacts the product's shelf-life, including promoting undesirable colors to the final product(Dorsa, 2008). Various impurities are naturally present in the seeds or originated from the harvesting and storage or formed during the extraction of crude oil and then after its refining(Abedi, Amiri, & Sahari, 2020).The bleaching process is one of the processes of oil refining that provides the steps to remove or reduce these impurities so that their deleterious influence on oil stability is reduced(Tavakoli, Sahari, Barzegar, & Ahmadi Gavlighi, 2019). The stage of clarification of vegetable oils requires a large amount of inputs, such as energy, adsorbents, and filtration aids, which represent a large part of the final cost of refined oil, with the optimization of their use a determining factor for the competitiveness of vegetable oil refineries. Since the optimization of the clarification process is an important factor, the present work aimed to raise with the specialized bibliography that aimed at the optimization of the process of clarification of different vegetable oils, as well as works that used unconventional and innovative technologies for obtaining clarified vegetable oils in an efficient and improved way.

Theoretical approach: Oils and fats extracted from oilseeds, fruits and almonds can vary from products with pleasant aromas and few impurities to products with a high content of impurities and sensory offensive odor. In this sense, the refining process aims to eliminate such impurities improving their physical, chemical and sensory characteristics for suitable human consumption(Wang, 2011).The final stage of the process of obtaining edible vegetable oils is the refining of crude oil, which consists of several physical and chemical steps aimed at removing impurities and one of its most delicate steps is the clarification where impurities from soybeans are removed through an adsorption process(Shahidi, 2005). According to Shahidi (2005)the term bleachingor clarification has traditionally been used to denote the pigment reduction process, such as pheophytins and carotenoids present in vegetable oils through the adsorption of these components in adsorbents known as bleaching earth or clarifying clay. After mixing with stirring and temperature control of the vegetable oil with the adsorbent to promote the removal of pigments, the adsorbent is removed from the vegetable oil through a filtration process and the resulting oil is cleaner and more stable. The bleaching process is a critical step in refining edible oil and can be considered one of the most expensive steps(Abedi, Sahari, & Hashemi, 2017).

The bleaching process occurs through several different mechanisms, including various chemical interactions. These mechanisms include (Aliyar Zanjani & Piravi Vanak, 2019):

1. Adsorption, the mechanism that captures impurities, occurs in three different ways: physics (with the involvement of Vander Waals' forces); chemistry, (by chemical absorption, with an electrochemical bond to the bleached earth surface); by molecular sieve (which, during filtration, retains impurities under pressure in the bleaching soil).
2. Filtration, the mechanism removing suspended or contamination to remove physically. The physical action of removing the suspended bleaching soil by simultaneous filtration removes the minor contamination absorbed by the bleaching earth particles.

The filters used in the bleaching process includeprocess filters (vertical and horizontal plate filters) and polishing filters (cartridges, plates). 3. Catalysis, the mechanism by which contamination is removed through interaction with the surface of the bleached earth. For example, the peroxides are effectively reduced by interaction between the bleaching earth and the oil (polymerized or decomposed into volatile products).

The adsorbent materials used in the clarification stage of vegetable oils are usually aluminum silicates, bentonites, activated carbons or montmorillonites that can be treated through an acidification process to increase the contact surface and, consequently, increase the adsorption power of the material(Moretto & Fett, 1989). The natural clarification earth, known as Fuller's earth, contains hydrated aluminum silicate. The most common natural bleaching adsorbent is the montmorillonite. Currently, instead of natural bleaching earth, bleaching adsorbents activated with acids (sulfuric acid and hydrochloric acid) is used. The amount of oil absorbed by the natural bleaching earth is 20-25% w / w, while for activated bleaching earth it is 35-40% w / w and for activated carbon it is more than 40% w / w(Aliyar Zanjani & Piravi Vanak, 2019).According to Gupta (2017)the impurities present in the oil to be clarified are attracted by attractive forces of Van der Waals with the active sites of the adsorbent agent and the force of attraction depends on factors such as the electrostatic force between the impurities and active sites of the adsorbent, the particle size of impurities, agitation of the mixing medium between the adsorbent and the oil to be clarified, porosity of the adsorbent and specific area of the adsorbent. The conventional process of clarifying vegetable oils using activated acid clays removes pigments, such as chlorophyll, among other undesirable components for human consumption, whether they are naturally present in vegetable oil or formed in the refining process(Boukerroui & Ouali, 2000). Among these, bioactive compounds such as tocopherols and sterols, which can cause loss of oxidative stability(Naz, Sherazi, & Talpur, 2011) and causing an increase in free fatty acids(Verleyen et al., 2002). The conventional procedure for clarifying vegetable oils generally leads to problems such as loss of oil entrained by the adsorbent, environmental problems and high costs of effluent treatments(Abedi, Sahari, Barzegar, & Azizi, 2015). According to Zschau (2001) the optimization of the clarification process depends on factors such as temperature, clarifying adsorbent dosage, reaction time and type of oil to be clarified. For O'Brien (2009) the key parameters for clarification are: procedure, dosage of adsorbent, type of adsorbent, temperature, time, humidity and filtration. In addition to these parameters, Gupta (2017)reports the concentration of soaps and phospholipids present in the oil to be bleached, formation of the homogeneous pre-layer on the filter plates, filter area and spacing between the filter plates as parameters to be controlled for an efficient bleaching procedure. Other technologies such as ultrasound bleaching(Abedi et al., 2015, 2017)or with the application of a high voltage electric field (HVEF)(Abedi et al., 2020; Abedi, Sahari, Barzegar, & Azizi, 2016) have been studied to clarify vegetable oils.

METHODOLOGY

The literature review took place between September 21 and November 23, 2020 through collections of materials in the database of the Coordination for the Improvement of Higher Education Personnel (CAPES), Web of Science, Medline Complete (EBSCO) and Scientific Eletronic Library Online (SciELO). We chose to list academic materials that considered the issues of optimization of oil clarification and oil bleaching optimization, submitted in national and international journals in the period between 2005 and 2020. As the objective was to verify results of the use of new technologies in the optimization of the process of clarification of vegetable oils, the criteria of choice were taken into consideration for the selection of the articles to be studied, in addition to works where the optimization of the process of clarification of oils using existing technologies but using new adsorbent agents and experimental designs that seek to optimize the clarification of vegetable oils.

ANALYSIS OF THE RESULTS

After surveying all the collected material, sixteen articles were obtained that fit the established selection criteria. These are studies carried out outside Brazil and bring new technology and optimization of the process of clarifying vegetable oils such as soybean oil, cottonseed oil, canola oil and rapeseed oil. Škevin et al. (2012)have sought to optimize the clarity of soybean oil through an experimental design of three factors: bleaching earth dosed (0.5, 1 and 1.5%), temperature (95, 105 and 115 °C) and reaction time (20, 30 and 40 minutes). A central composite design to determine the parameters that significantly interfere with clarification, having as answer the color and transparency parameters of the clarified oil and parameters related to the oxidative stability of the clarified oil. With the result of their experiments, the authors found the proportionality between the dosage of bleaching soil and the efficiency of pigment removal, as well as the negative effect of increasing the temperature in the clarification process and the increase in second-order oxidative compounds. The most suitable condition for the bleaching of soybean oil was given in the clarification dosage using 1% of clarifying clay in relation to the mass of the oil to be clarified at 95 °C for 20 minutes. This process condition proved to be efficient for reducing color and not generating degradative compounds, however, it was not the best option when it comes to the content of tocopherols present in the clarified oil. In another experiment, Saneei, Hossein Goli, & Keramat (2015)investigated the optimization of the canola oil clarification process using an experimental design involving three factors at three levels and an intermediate factor. The factors employed were temperature, clarifying clay dosage and time, with responses measured and analyzed using the response surface methodology. The answers were color parameters and chemical parameters that measure the oxidative stability of the clarified oil. Through the experiments carried out by Saneei et al. (2015) the authors could conclude that the clarification process was more efficient when performed for 42.46 minutes at 110ºC with the addition of 3% of clarifying clay in relation to the mass of the oil to be clarified. The authors cite that the adsorbent material used outside sepiolite and that it has a low cost when compared to commercial adsorbents such as bentonite, however, does not conclude whether the replacement of bentonite by sepiolite dosed in 3% is financially viable. In previous studies, Sabah & Çelik (2005)investigated the use of sepiolite of Turkish origin in canola oil and obtained that the maximum yield occurred when 1.5% of the adsorbent was dosed at a temperature of 100 °C. Nassar, Abdelrahman, Aly, & Mohamed (2017) reported the preparation of nanostructures of zeolite using a low-cost hydrothermal treatment of silica gel, aluminum nitrate and sodium hydroxide. The zeolite of mordenite produced were applied to purify crude soy oil of yellow and red colors. And they were also used to remove methylene blue dye from wastewater. The kinetic data showed that the dye adsorption process followed pseudo-first order models, diffusion, liquid film diffusion and pore diffusion, while the step of determining the adsorption rate is controlled only by the pore diffusion model.

Studies have reported that clarifying clays are expensive and cause an increase in oil acidity due to the conversion of triacylglycerol's to free fatty acids(Abedi et al., 2016; Hussin, Aroua, & Daud, 2011). A study was conducted to use sand powders and sea shells as natural materials to reduce the color of crude soy oil compared to synthetic bleaching earth during the refining process(Aly, 2018). In this work, some chemical properties were studied, such as, values of acid, peroxide and thiobarbituric acid, and the effect of bleaching earth treatments, sand powders and shells, on the fatty acid profiles of crude soybean oil. The results showed that the red color removal of crude soybean oil treated with sand powders and shells was greater than the use of bleaching earth and the greatest removal was observed with shell powder. Shell and sand powders significantly increased ($p \leq 0.05$) the values of acid, peroxide and thiobarbituric acid in crude soybean oil. Mathematical models have been proposed to describe the bleaching process of soybean oil and the function of its parameters (temperature: 80-120ºC; clay dosage: 0.25-2%; contact time: 10-30 min)(Henache, Boukerroui, & Kashi, 2018). The models were developed by means of multiple linear regression analysis (MLRA) and were performed using the Matlab programming language. The optimal conditions in this soy oil bleaching study were temperature 100 ºC, 2% clay dosage w / w and contact time 30 min. The highest bleaching capacity found was 81.04% at 426 nm, 90.60% at 451 nm and 93.66% at 479 nm. The developed models made it possible to predict the bleaching capacity representing the removal of β- carotene and chlorophyll-a pigments present in crude soybean oil at each λ max. They also allowed better control of the most influential parameters in the bleaching stage and contributed to the optimization of the bleaching clay tailings by optimizing the amount of clarifying clay used in the refining process, thereby reducing the risks of pollution. Ghorbanpour (2018)studied the process of bleaching soybean oil using commercial bentonite and a combination of iron oxide / bentonite. The alkaline ion exchange process increased the surface area of the bentonites. The experimental results indicate that the composite prepared for 1 min showed the same efficiency in bleaching crude soy oil with bentonite. Thus, bentonite / iron oxide can efficiently decolorize crude soybean oil by removing coloring agents such as β-carotene. The author concludes that this process provides a good adsorbent with better bleaching properties than commercial bentonite.

The use of calcium sulfate dehydrates ($CaSO_42H_2O$) as a bleaching agent during the refining of crude soybean oil was investigated byChakawa, Nkala, Hlabangana, & Muzenda (2019).Different proportions of the bleaching agent $CaSO_42H_2O$ in relation to the crude oil (2, 4, 5, 10, 20 and 30% by weight) were prepared. Each of the 7 samples of the crude oil was preheated to a temperature of 80ºC - 120ºC. The results indicate that calcium sulfate dehydrate ($CaSO_42H_2O$) is an effective, ecologically correct, cheaper, and effective bleaching agent in removing red color while maintaining the content of free fatty acids. Considering the importance of bleached earth with activated carbon in reducing impurities in vegetable oils, the study byAliyar Zanjani & Piravi Vanak (2019)was carried out with the objective of investigating the effects of the bleaching process on the physicochemical properties of bleached soybean oil. The bleaching process was carried out with bleaching soil (1% w / w) containing different concentrations of activated carbon (0.1% to 0.5% w / w). The activated coal is highly effective in the separation of green pigments and polycyclic aromatic hydrocarbons (PAH 's). The PAH are among the most toxic compounds in edible oils. Fatty compounds are joined by several aromatic rings, including hydrogen and carbon atoms. These compounds are also semi-volatile or non-volatile, non-degradable, highly organic, stable and toxic to the environment (Singh, Varshney, & Agarwal, 2016; Stenerson, Shimelis, Halpenny, Espenschied, & Ye, 2015). The weigh d the high whitening capacity of the activated carbon, it is not used alone because of its high cost, but generally are combined 10-20 parts by bleaching earth with a weight of activated carbon.

The results of Aliyar Zanjani & Piravi Vanak (2019)indicated that the bleaching process did not affect the oil structure, while the percentage of free fatty acids (FFA) in the oil samples after bleaching was increased compared to white. During bleaching, the peroxide values of the samples and color decreased. Since vegetable oils are proven to be the main sources in the diet, refining, especially the bleaching process, plays a decisive role in the production of high-quality oil. Therefore, this step is one of the critical control points (CCP) to guarantee the quality of the final product. The use of bleached earth with activated carbon to investigate the reduction of PAHs in soybean oil was also investigated in the work of Aliyar-Zanjani, Piravi-Vanak, & Ghavami (2019). The bleaching process was carried out with different amounts of bleaching earth (1% w / w) and activated carbon (0.1% to 0.5% w / w). HPLC with fluorescence detector was used to determine the PAHs in oil samples after undergoing extraction and cleanup procedures. The results of linearity indicated that there was a linear response with high linear regression coefficients of determination for all 4 PAHs analyzed. ($R^2 > 0.9950$). In addition, the recovery percentage was calculated from 83.8% to 106.2%; LOD (limit of detection) and LOQ (limit of quantification) were 0.06-0.2 μ gkg^{-1} and 0.2-0.61 μ gkg^{-1}, respectively. An analysis of the PAH contents indicated that the bleaching process, including an application of 0.27% to 0.5% w / w of activated carbon, led to the elimination of the PHA content. Vegetable oils have been shown to be the main sources of HPAs in the diet, the industrial use of activated carbon during the bleaching of vegetable oils is highly recommended. In the study by Deniz, Bilici, & Tuncer (2020) the parameters of the bleaching earth system were investigated experimentally for a factory that processes soybean and sunflower oil. The amount of bleaching earth used, and the bleaching time were used as independent parameters, and the values of color, acid and number of peroxides were determined. The results showed that the proportion of bleaching soil and the bleaching time had a prominent effect on color, but their effects on acid and peroxide numbers were limited. There was no linear relationship between the bleaching time and the color change, and the excess time had a negative effect on the bleaching process depending on the amount of bleaching soil.

The application of ultrasound in food processing is a new and interesting technology, when compared to traditional chemical and physical methods of oil extraction(Abedi et al., 2017). Ultrasonic waves are sound waves with frequency above the human hearing capacity and with sufficient intensity to promote cavitation in the environment where it spreads(Wu, Zivanovic, Hayes, & Weiss, 2008). In turn, the phenomenon of cavitation is characterized by the rapid collapse of micro cavities (gas bubbles, steam or air) that promote various physical and chemical changes in the environment in which they occur, such as changes in the pressure of the medium, shock waves caused by release of energy and release of free radicals(Su et al., 2013). The use of ultrasonic waves in the oil extraction process brings great advantages when compared to traditional chemical processing, with reduced process time, reduced energy consumption(Chandrapala, Oliver, Kentish, & Ashokkumar, 2012)and increased yield without significant changes in the profile fatty acids in the raw material(Zhang et al., 2008). The experiments by Su et al. (2013)aimed at the use of ultrasonic technology in the clarification of rapeseed oil in the face of variations in the conditions of the clarification process such as different adsorbents, dosage of adsorbent, temperature and amplitude of the ultrasonic wave, having as a response the measurement of pigments by means of spectrophotometry, setting the wavelength at 446 nm. As reported by Su et al. (2013) the clarification using ultrasonic waves is the result of the adsorption and degradation of pigments present in the oil, such as chlorophyll and carotenoids. When employed low - frequency ultrasonic waves, the adsorption of pigments results in reduction of absorbance measured at 446 nm of 1,82 to 0,65. When employed high frequency ultrasonic waves, reducing the absorbance measured at 446 nm is 1,82 to 0,74, resulting in pigment adsorption and degradation caused by the effect of ultrasonic waves at high frequencies. His studies indicate that the clarification of rapeseed oil is best efficient when applied 40% of the maximum ultrasonic range and using bentonite as an adsorbent. It was also found that, with the increase in ultrasonic amplitude, the removal and degradation of pigments is accentuated. Still in the work of Su et al. (2013) after clarification using ultrasonic waves, an increase in primary oxidation compounds was found, however, secondary lipid oxidation compounds remained constant, making the rapeseed oil clarification through the use of ultrasonic waves an alternative to the process conventional clarification.Abedi et al. (2016)found a decrease in primary oxidation compounds, such as the peroxide index, both in samples submitted to conventional clarification and in samples submitted to ultrasonic clarification, however, the values of peroxide index in samples submitted to the first process showed greater results than in samples submitted to the second process.

In their work, Abedi et al.(Abedi et al., 2015)evaluated the impact of factors such as ultrasonic amplitude (0, 45%, 90% of amplitude) temperature (35, 45, 55 and 65 °C), adsorbent dosage (0.5, 1, 1.5 and 2%) and time of reaction (10, 15, 20, 25, and 30 minutes) in the clarification process and, through his experiments, obtained that the use of ultrasonic waves in conjunction with adsorbents aiming at the clarification of soy oil caused reductions in the concentration of chlorophyll present in the oil, around 94.66% and 95.25%, when using a frequency of 20.4 Hz and 15.6 Hz, respectively. Through the study,Abedi et al. (2015)were able to verify that the combined process of ultrasonic waves and clarifying clay for the clarification of soybean oil, since optimized, can reduce the process temperature by 35%, adsorbent dosage by 35% and time by 10%, when compared to clarification using only the adsorbent, and that the increase in ultrasonic amplitude tends to increase the removal of bioactive compounds, such as tocopherols. Abedi et al. (2017) investigated the reduction of color (red and yellow) and pigments (caroten the go and chlorophyll) from soybean oil by treating ultrasonic under different conditions (gas temperature –50°C and 65 °C, time - 17:23 min, frequency 25 and 40 kHz and type of ultrasound - bath or probe) and combination of these factors. Results showed that the combination of ultrasound technology with different gases with clay fining increased the efficiency of mass transfer and cavitation d pigments in clay surface, enhancing the bleaching process as compared to conventional bleaching process. To increase the pigments removal, the lowest frequency (25 kHz) was more efficient than the highest frequency (40 kHz). The ultrasonic bleaching process was more efficient in removing chlorophyll carotene compared to the FDI, and is therefore suitable for soybean, olive, and canola. Gas was the most important factor in reducing oil pigments and colors, compared to other factors. The most effective gases were in decreasing order: argon, air, helium, and nitrogen. However, temperature and frequency can alter the effect of gases during the ultrasonic bleaching process. The authors conclude that, to reduce the temperature and time consumption in the bleaching process, ultrasonic bath systems under gas jet are recommended. Abedi et al. (2016)performed an innovative experiment was applied a high voltage electric field (HVEF) to optimize the clarification process of vegetable oils. HVEF is one of the main non-thermal processing technologies. For such evaluation, an experimental planning was carried out where the effect of process parameters was evaluated, such as adsorbent dosage (0.5 to 2%), voltage (0 to 24 kV), distance (8 to 12 mm) number of cathodes and anodes (4 to 8) and variations in dosages of hydrochloric acid (0 to 50 mM). Through their experiments, the authors measured the color parameters and physical parameters that indicate the oxidative state of the clarified oil in response, obtaining results that demonstrated that the use of high voltage electric fields in the soy oil clarification process could reduce by 50% the dosage of adsorbent, 28% the temperature of the process and 35% the reaction time and 66% the dosage of adsorbent, 28% the temperature of the process and 35% the reaction time, when applied in the clarification of sunflower, proving to be an innovative and efficient alternative to the conventional clarification process.

In the study by Abedi et al. (2020) the amounts of trace elements (Fe (II) and Cu (II)) and pigments (chlorophyll and carotenoid) in soybean oil were evaluated under the high voltage electric field (HVEF) bleaching method at different voltages (10 and 20 kV), temperature (35–65 °C), time (0–30 min) and clay percentage (0.5–2%) and then were compared to the industrial bleaching method (IBM). The thermodynamic parameters (ΔG °, ΔH ° and ΔS °) showed that the adsorption of trace metal ions and pigments in bentonite clay under IBM and HVEF was viable, endothermic, and spontaneous between 35 and 65 °C. The results indicated that the HVEF, especially at higher voltages, has a remarkable ability to remove metal ions and pigments from soy oil compared to IBM. The authors further revealed that HVEF has other advantages over IBM that include 50% reduction in clarifying clay, 35% reduction in processing time and 28% reduction in temperature resulting in greater bleaching efficiency and energy consumption. The studies specify in detail the experimental procedure adopted and present the results always aiming at optimizing the clarification process. The most studied parameters are the dosage of soil, temperature, and reaction time, which effectively demonstrate a direct impact on the optimization of the process, being variable depending on the raw material studied. The use of new technologies, such as ultrasonic waves and high-voltage electric fields, aiming at the higher yield of soy oil clarification, has great potential, since it promotes a great reduction in process parameters.

Final considerations

The presence of impurities in the oil causes darkening, foam, reduced smoke point and oxidative stability, along with other adverse effects during the process, which eventually reduce the safety and quality of the finished product. The optimization of the vegetable oil clarification process is determined by combining the factors reaction time, reaction temperature and addition of adsorbent, these variables being specific to the application carried out. The use of new technologies shows a promising aspect, since there is a large reduction in inputs causally linked to the costs of the clarification process, which makes such technologies highly attractive. The use of ultrasound in the clarification of vegetable oils has numerous advantages, such as shorter processing time, low energy consumption, improved extraction performance due to the use of alternative solvents, improved extraction of heat-sensitive components, greater yield due to the increase of the extraction yield and increase of the aqueous extraction yield without the use of solvents. In addition, the equipment used is relatively cheaper than other technologies. The weigh the advantages presented technology using ultrasound there is no effective proof of the cost reduction front to optimize the clarification process. The fact that only works by foreign authors are found indicates that study and innovation in the process of clarifying vegetable oils is very scarce in Brazil, since research in this area is almost predominantly carried out by the private sector. The strengthening of the alliance between the private sector and the academic environment results in works of relevance such as those reported in this bibliographic review, which positively impacts all those involved.

*The authors declare that there is no conflict of interest.

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