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International trends in intellectual property on extraction methods and technological applications of coffee oil

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ABSTRACT: This work aimed to present the current scenario of technological innovation involved in extraction and applications of coffee oil through patents listed in Derwent Innovations Index and INPI platform. Keywords combinations such as (i) «coffee oil» AND «extraction»; (ii) «coffee oil» AND «aroma» (iii) «coffee oil» AND «microencapsulation», (iv) «spent coffee grounds» AND «coffee oil» and (v) «coffee oil» AND «spray drying» were used as search terms on Derwent's database, which resulted in 62 patents. Of those 62 patents, 80 % of them were classified as A23 – (food products and their processing, coffee aroma; coffee oil, etc.), 31% were granted in the years between 2014 and 2016, of which USA leads with 27.9% and 70 % of the patents. In both cases, the supercritical CO2 technique was applied to oil extraction. Nestec S.A., a subsidiary of NESTLÉ, at 14.5 %, holds the majority of active patents. Of the patents analyzed on the INPI platform (the same Keywords combinations are used, except in portuguese), the most uses of coffee oil were related to the production of instant coffee, pharmaceutical manipulations and polymer matrices. Pressing was the extraction method that represented 54 % of the 12 patents found. The main classification was C11B with 52 % of patents. Microencapsulation technique to preserve aroma and bioactive compounds has been used in some of patents. This topic proved to be promising in terms of commercial exploitation due to its efficiency in preserving compounds from vegetable oils. The national industry and the academic community must be attentive to the coffee sector that influences the Brazilian economy, observing opportunities for development and technological innovation.

KEYWORDS: Coffee oil, extraction, intellectual property, microencapsulation, spray drying.

1 BACKGROUND

Coffee is the world's leading commodity amongst agricultural products. Brazil is by far the largest producer and exporter of coffee beans and accounted for 36 % of the world's production [1]. In 2018, Brazil produced 61.65 million bags (of 60 Kg) of coffee, an increase of 37.1 % compared to 2017, of which the country exported 35.2 million bags, reaching 5.09 billion dollars with an average price of US$ 144.53 per bag [1]. Furthermore, Brazil is the second largest coffee consumer on the planet, surpassed only by United States [2]. The coffee produced and consumed both domestically and abroad generates a high income, also results in generation of Spent Coffee Grounds (SCG), an important by-product of the agribusiness. It is estimated that in 2020, the coffee produced in Brazil will achieve about 2.4 million tons of SCG, considering that processing of one ton of coffee generates approximately 650 kg of SCG [2].

SCG consists of proteins, carbohydrates, ashes, pigments, fibers, lipids and also unsaponifiable matter, which has high concentration of important bioactive compounds with antioxidant, antimicrobial, anti-allergenic, anti-atherogenic, anti-inflammatory and anti-thrombotic properties [3], [4]. The presence of these compounds, which have beneficial effects on human health, makes spent coffee grounds a potential source of natural antioxidants and has motivated several studies [4], [5], [6], [7], [8] and [9]. Since it contains approximately 20 % of lipids, coffee beans has attracted greater attention in the world market, due to the wide application of coffee lipids in the food and cosmetics formulations.

Considering the volatile profile identified in the lipid fraction of roasted coffee oil, it is potentially applicable in the food industry as a flavor enhancer due to its pronounced aroma [4], [10], [11]. The lipid fraction of coffee has approximately, 75 % triglycerides and up to 19 % of unsaponifiable matter. This last fraction is composed mainly of terpenic esters, tocopherols and sterols, in addition to polar waxes and lipids [12]. The main fatty acids described by Folstar et al. (1985) [13] for coffee oil of the species Coffea arabica are linoleic acid (41.2 to 42.6 %), palmitic acid (35.2 to 36.7 %), oleic acid (9.5 to 11.9 %), stearic acid (7.2 to 9.7 %), linolenic acid (1.3 to 2.7 %) and arachidonic acid (0.3 to 1.5 %). Roasted coffee oil has a peculiar aroma and nutraceutical properties, it's use in food depends not only on its chemical characteristic but also on its physical properties.

Coffee oil can be obtained from green, roasted beans or SCG through classical mechanical process (pressing) or chemical process (solvent extraction) [14]. Regardless of the method used, the lipid extracts obtained contain volatile substances (characteristics of the aroma), phenolic compounds which exhibit remarkable antioxidant properties and substances with anti-ultraviolet activity [19]. Technological changes in the edible oil extracted are mainly aimed at stabilizing bioactive and volatile compound, which are essential to guarantee the use of coffee oil as an ingredient for several expanding sectors [15]. Furthermore, various environmental factors have adverse effects on the stability of micro bioactive compounds and fatty acids present in the coffee oil, hence the microencapsulation of coffee oil has been evaluated by many researchers as one of the techniques that offers protection against oxidation from external factors such as heat, humidity and light [15], [16].

The protective activity of the wall materials used in the microencapsulation process reveals the potential application of this technique to preserve the volatile compounds of coffee oil, increasing the useful life, especially the aromatic compounds [16]. The use of agro-industrial by-products, in particular SCG, as a source of edible oil and other compounds such as phenolics, diterpenes, sterols, among others, in addition to substances characteristic of the aroma, arouses scientific interest, due to the availability and potential of this residue. In this context, the objective of this work was to identify the main international trends in the process of extraction and use of coffee oil with a view to its use in the food and pharmaceutical industry through an analysis of deposited patents.

2 MATERIAL AND METHODS

The technological research was carried out from April to July 2019 between 1963 and 2019. This period was therefore taken into account to evaluate the patents listed in the Derwent Innovations Index® (Clarivate) and National Institute of Intellectual Property Office (INPI, acronym in portuguese) databases. The following keyword combinations were used in the topic field of the databases: (i) "coffee oil" AND "extraction"; (ii) "coffee oil" AND "aroma" (iii) "coffee oil" AND "microencapsulation", (iv) "spent coffee grounds" AND "coffee oil" and (v) "coffee oil" AND "spray drying". English keyword combinations were used on the Derwent's database, while the equivalent Portuguese keywords were used to search for patents listed at the INPI database. Results with the aforementioned keywords present in the patent's title and/or in the abstract were considered for analysis. The patent documents found were collected, treated and analyzed for information about the technological survey. Those international classification codes were considered significant for this work, which presented a quantity above 5% of representativeness. It is to be noted that, the search priority was not given to the patents that are within the secrecy period of eighteen months, i.e. only the already published patents considered. The research focuses on the collection of data about the main extraction techniques and applications of extracted oil from green or roasted coffee. Bibliometric treatment was performed on the collected data by Microsoft® Excel software version 2010, according to year of application, applicants (companies, institutions or individuals), areas of knowledge, countries of origin and codes of the International Patent Classification (IPC).

3 RESULTS AND DISCUSSION

The search on the Derwent Innovations Index platform resulted in a total of 67 patents referring to the technologies associated with the product of interest (Figure 1).

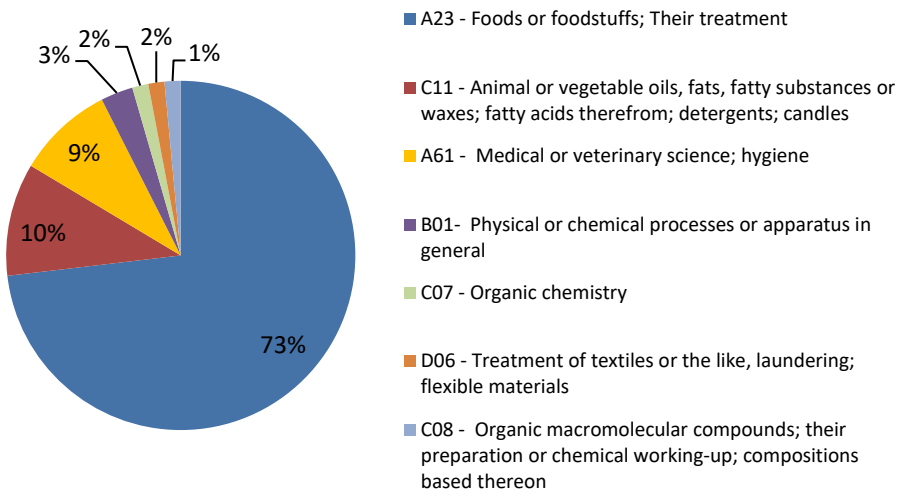

Fig. 1. Distribution of patent according to IPC codes

Coffee oil has been gaining popularity in the world market, since it has substances that meet the industry's demand for natural compounds. Amongst the patents analyzed, several applications of coffee oil were observed, highlighting its use for flavoring beverages [17]. In addition to that, other applications have been observed, such as the production of polyol from the epoxidation reaction for producing polyurethane fibers, extraction of bioactive compounds such as chlorogenic acid and kahweol [18], besides compounds with anti-ultraviolet activity (anti-UV) [19] for cosmetics formulating and also microencapsulation of volatile compounds [20].

In this work, as a first step, the patent applications were organized according to the International Classification. In the the filtered classifications are shown, which takes into account of the section and its class. This division followed the provisions of the IPC's, as established in the Strasbourg Agreement, in 1971, with last update on 12/21/2012. After this classification, a selection was applied for the data collection directed to this work for patent applications involved with the theme of "main uses and technologies for extracting coffee oil".

The following patent sections were excluded: physical or chemical products or devices in general (B01), organic chemistry (C07), organic macromolecular compounds (C08) and treatment of textile products, instrumentation (D06), as they represented less than 5 % of patents analyzed and did not fit the objectives of the mapping carried out. After the re-classification, 62 patents were considered for analysis, with codes belonging to the sections A23F, A23L, A61K and C11B for this work (Figure 2).

According to Figure 2, most patents were classified in the class A23F, which represents 70 % of the documents analyzed, in the area of food products like coffee, tea, their manufacturing, processing, physical treatment and conservation. The second class with the highest number of patents was C11B, representing 11 % of production, which comprises of raw material extraction from waste substances, refining or preserving oils, fatty substances such as lanolin, fatty oils or waxes, essential oils, perfumes (drying oils) and production of oils or fats from various raw materials. The code A61K focuses patents on preparations for pharmaceutical products and A23L on the methods of preparation, modification, preservation of food products and soft drinks, both codes constitute 6 % of the total patents analyzed. From the initial analysis of the patent classes, it shows a clear trend in the application of raw materials with sensory and bioactive properties in the food and cosmetics industries.

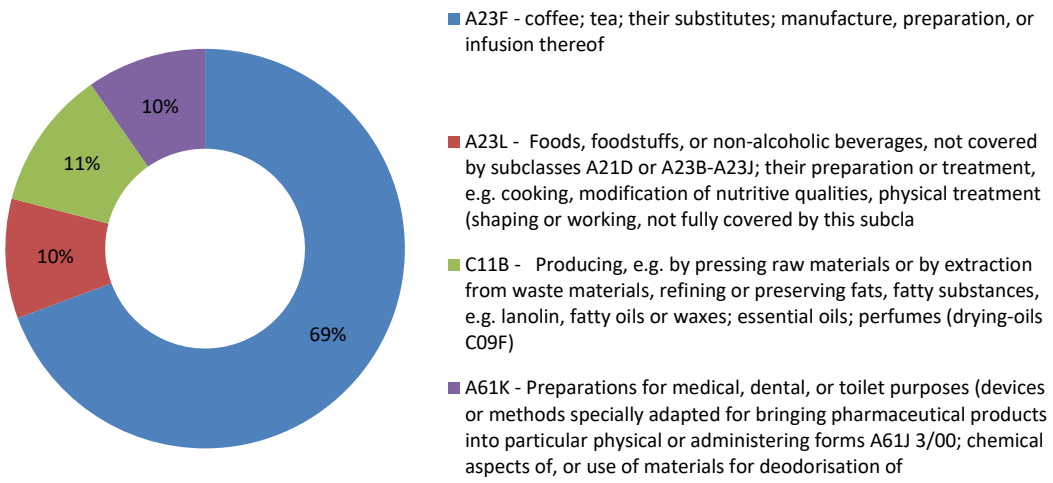

Fig. 2. Most prominent IPC codes in the survey carried out on the Derwent base

Since majority of patents were categorized under IPC A23F, the subdivisions within this class were analyzed (Figure 3). Out of 43 patents under classification A23F, the code A23F-005/46 represented 21 % of the patents researched and it presents by definition works with coffee aroma, coffee oil, flavoring of coffee or coffee extract synthetic coffee flavorings. Code A23F-005/48 - Isolation of coffee flavoring or coffee oil - represented 19 % of patents, followed by code A23F-005/40 - Coffee infusion, aroma extracts of coffee or coffee oil - with 9 % of the patents under the A23F classification.

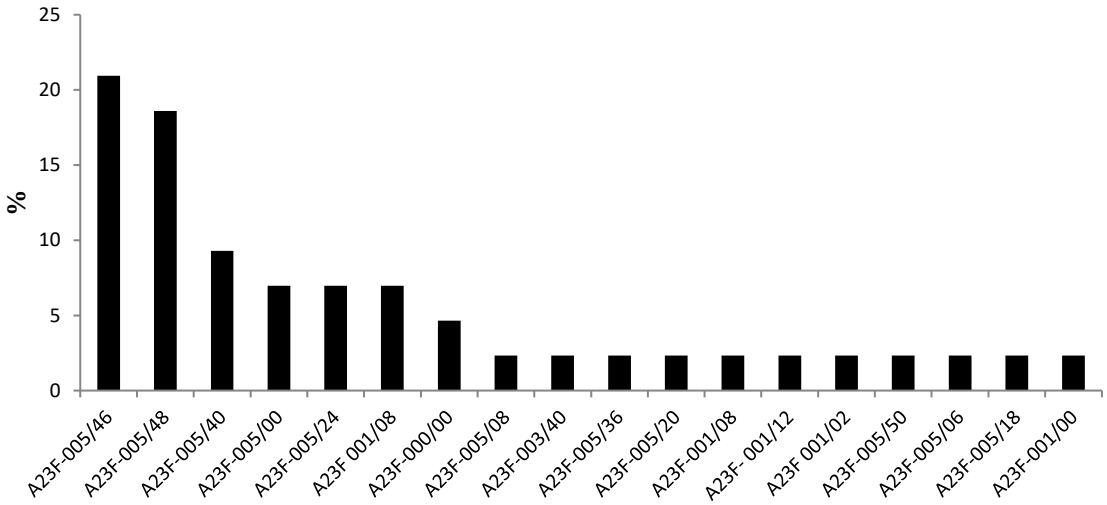

Fig. 3. Main codes of International Patent Classification with filter in A23F

A23- FOODS OR FOODSTUFFS; THEIR TREATMENT, NOT COVERED BY OTHER CLASSES; A23F- COFFEE; TEA; THEIR SUBSTITUTES; MANUFACTURE, PREPARATION, OR INFUSION THEREOF; A23F- 005 - Coffee; Coffee substitutes; Preparations A23F 005/46 - Coffee flavour; Coffee oil; Flavouring of coffee or coffee extract (synthetic coffee flavours A23L 27/28); -A23F 005/48- Isolation of coffee flavour or coffee oil; A23F-005/40 - using organic additives, e.g. milk, sugar; A23F-005/00- Coffee; Coffee substitutes; Preparations thereof; Extraction of coffee (isolation of coffee flavour or coffee oil A23F 5/48); Coffee extracts (with reduced alkaloid content A23F 5/20); A23F-005/24- Making instant coffee (methods of roasting extracted coffee A23F 5/06); A23F 001/08; A23F-000/00; A23F-005/08- Methods of grinding coffee (coffee mills A47J 42/00); A23F-003/40- Tea flavour; Tea oil; Flavouring of tea or tea extract (synthetic tea flavours A23L 27/20); A23F-005/36- Further treatment of dried coffee extract; Preparations produced thereby, e.g. instant coffee (removing unwanted substances A23F 5/18; flavouring A23F 5/46); A23F-005/20 - Reducing or removing alkaloid content; Preparations produced thereby; Extracts or infusions thereof; A23F-001/08; A23F-001/08; A23F- 001/12; A23F 001/02; A23F-005/50 - from coffee extract; A23F-005/06- Treating tea before extraction (reducing or removing alkaloid content A23F 3/36); A23F-005/06- Preparations produced thereby (tea extract preparations A23F 3/16); A23F-005/18 - from coffee extract; A23F-001/00.

The annual evolution of patent filings related to coffee oil, in the different industrial sectors (extraction, application in food and cosmetics) between the years 1970 and 2019 is shown in The period between 2014 and 2016 had maximum number of patents filed (31 %) of all existing patents in the Derwent Innovations Index database, considering the keywords applied in this research.

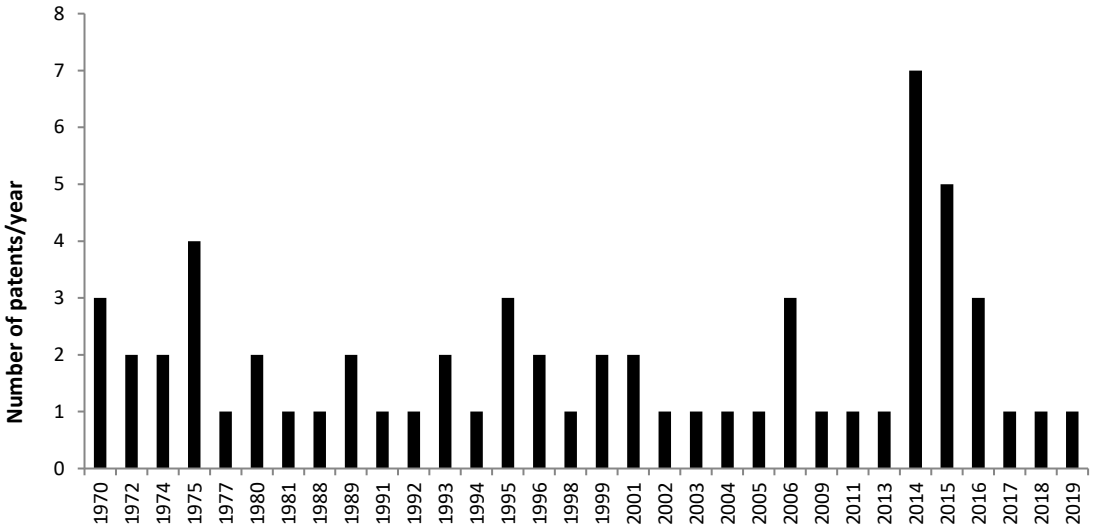

Fig. 4. Evolution of patent filings between 1970 and 2019 in the Derwent database

Among the 62 patents considered, the main countries with patent applications can be seen in The United States leads with 17 patents (27.9 %), followed by Japan with 13 (21 %). 8.2 % (5 requests) were applied by European countries through EP- European Patent Office.

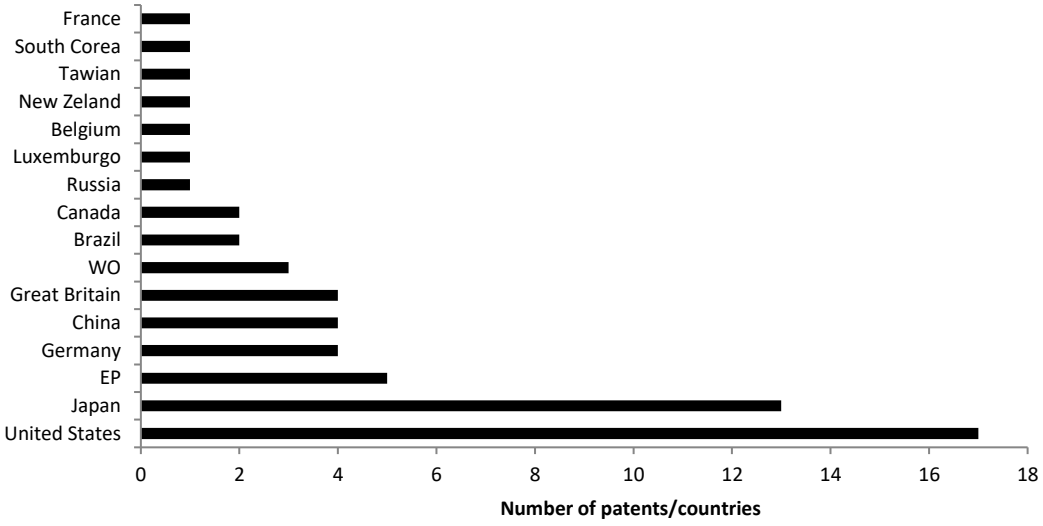

Fig. 5. Number of granted patents/countries listed in the Derwent database

In order to obtain coffee oil rich in chemically active components, the extraction method plays a crucial role in the process. Therefore, patents were assessed for the method of extraction and selection of the solvent used in the case of chemical extraction. For the purpose of recovering coffee oil, amongst the patents analyzed the main technologies involved were mechanical pressing, supercritical fluid with CO_2, use of volatile solvents (mainly with hydrocarbons such as heptane, ethanol, acetone, ethyl acetate, chloroform and ether of oil) and ultrasonic waves. It is worth noting that 70 % of the patents refer to the use of supercritical CO_2 as extraction technique. Some works used the combination of two or more techniques.

The patent, CN107674758-A – *"Extraction of coffee oil by ultrasonic subcritical fluid extraction involves extracting of dried coffee using solvent, ethanol in general, under preset conditions, removing the solvent and residue using thin-film evaporator"* [21], describes the extraction technique of coffee oil from SCG, using fossil solvents, supercritical fluid and ultrasonic waves. The solvents used were liquid propane, butane, isobutene and dimethyl ether. This combination of techniques enabled the effective and complete extraction of the active components of coffee, with satisfactory yield. The solvent can be recycled efficiently, thereby reducing solvent loss and operating costs. The product obtained did not contain solvent residue and can be used without restrictions in the food and cosmetic industries.

Even though ethanol, produced from biomass, with a lower toxicity as compared with fossil solvents, only one patent addressed this as a solvent to obtain coffee oil [22]. The interest in ethanol as solvent in the extraction processes is due to the fact of recovering an extract more rich in phenolic, terpenes and aromatic compounds, generating a fraction rich in natural antioxidants from a more sustainable technology [4], [23]. In addition to oil extraction, the supercritical fluid with CO_2 technique has also been patented for extracting aromas present in coffee oil and incorporation into other vegetable oils [24]. The microencapsulation of vegetable oils is observed as a worldwide trend in the preservation of aromatic and bioactive compounds (16).

Microencapsulation of coffee oil has been described in WO2010038064-A1-*"Consumer care or food composition e.g. toothpaste, consists of mesoporous microparticulate material, at least some of pores of which are loaded with ingredient, where mesoporous microparticulate material is encapsulated by capping layer"*. The invention was intended for the use of coffee oil in the food industry [25].

The aforementioned patent suggests that the use of microencapsulated material (coffee oil) in food formulation provides high availability of bioactive ingredients, due to the ability to control and direct their release, besides protecting unstable compounds from oxidative reactions. The microencapsulated material applied in the food composition increases the useful life of the compounds responsible for the aroma, without necessarily requiring the use of natural essential oils or the use of other ingredients that can negatively affect some properties of the food [16], [26]. In this sense, the technique of microencapsulation of vegetable oils has been recommended to prevent the loss of volatiles, the degradation of lipids, unwanted interactions with other ingredients, etc.

The microencapsulation process produces microparticles formed by the oil (located mainly in the core) surrounded by the wall material (encapsulating material). This process not only prevents losses and chemical changes during the production and storage of food, but also results in a versatile ingredient in powder form and with new technological properties [12], [16], [26]. Given the small number of patents related to microencapsulated coffee oil and the wide applicability of this product, it is possible to confirm that there is still a vast area to be researched and that new patents can be developed, enhancing the technological innovation of these materials and their applications.

Among the 62 patents analyzed, 48 documents belong to a group of the 13 main applicants (Table 1), ranging from 2 to 9 patent applications per applicant. The main applicant was Nestec S/A, a Nestlé subsidiary with 9 orders. It was possible to verify that among the main actors in the process, there was no university or government research institutes Therefore, most applicants belong to the private sector, such as General Foods Corporation, Ajinomoto General Foods Inc. and Kraft Food Corporation, etc.

Table 1. Main Applicants*

Applicants	Numbers of patents
NESTEC SA	9
GENERAL FOODS CORP	6
PROCTER & GAMBLE CO.	6
SOC PROD NESTLE SA	6
UCC UESHIMA KOHI KK	4
AJINOMOTO GENERAL FOODS INC	3
KONINKLIJKES DOUWE EGBERTS B.V.	3
KRAFT FOODS RD INC	3
HAIR ORIGHT INT CORP	2
NESTLE SA	2
SINGTEX IND CO LTD	2
ZELLER B L	2
Total	48

**Companies that filed only one patent were not listed*

The company Resitec, from Brazil, has 1 patent filed with the researched theme, related to the process of obtaining unsaponifiable material from the coffee oil for sterols and terpenes recover [27]. Another invention of Brazilian origin listed in the Derwent database belongs to the Regional Cooperative of Coffee Growers of Gaxupé and has been patented for oil extraction by pressing. According to the authors, this process is a low cost and resulted in a high-quality product, naturally free of solvents [28]. Despite occupying the first position in production and the second in consumption of coffee in the world [2], the number of Brazilian patents worldwide related to this theme is still very small. Only twelve (12) patents were found on the INPI platform in the search. From these, 50 % belong to the C11B class, followed by A23F (42 %) and A61K (8 %). In Figure 6, the patent codes filed on the Brazilian platform are listed.

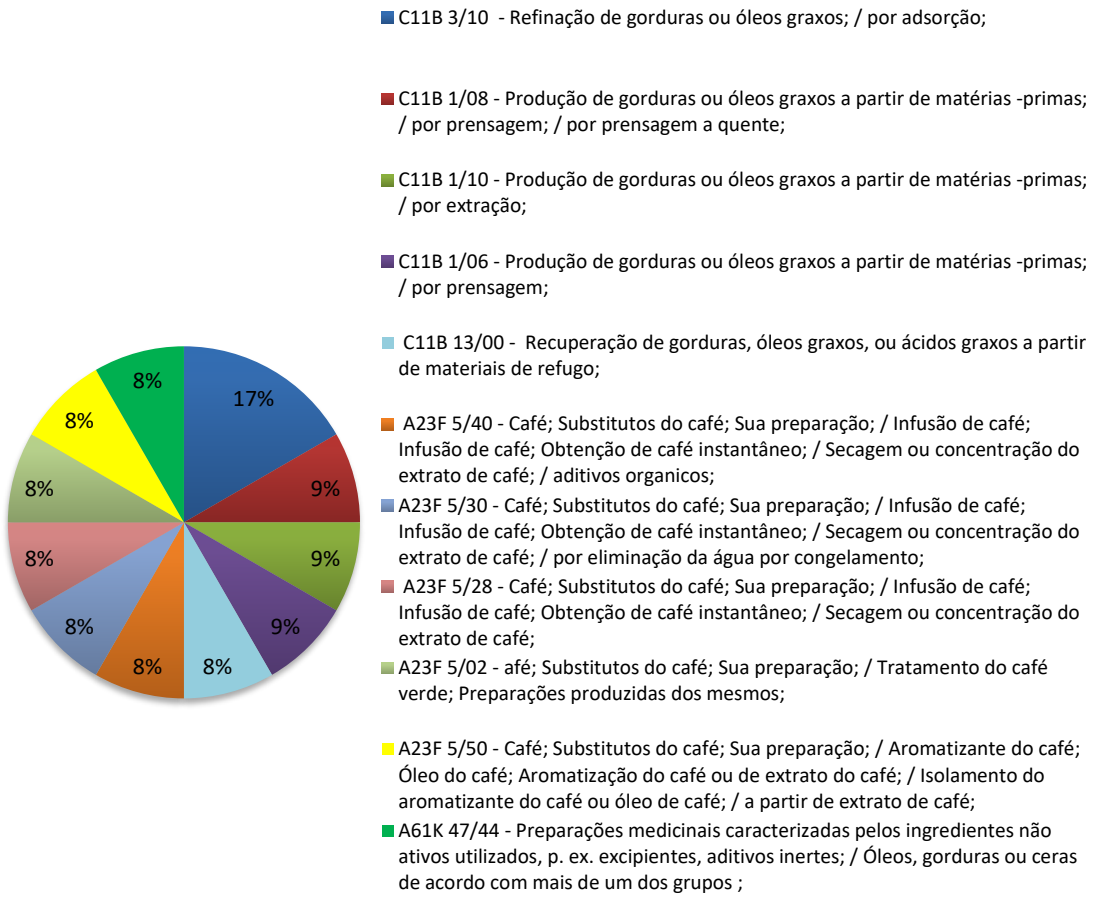

Fig. 6. Distribution of IPCs related to obtaining coffee oil by international classification codes on the INPI database

Within the INPI, a survey was carried out on the raw materials related to the keywords applied, identifying which were most representative on the Brazilian platform. shows the raw materials used to obtain oil in these patents.

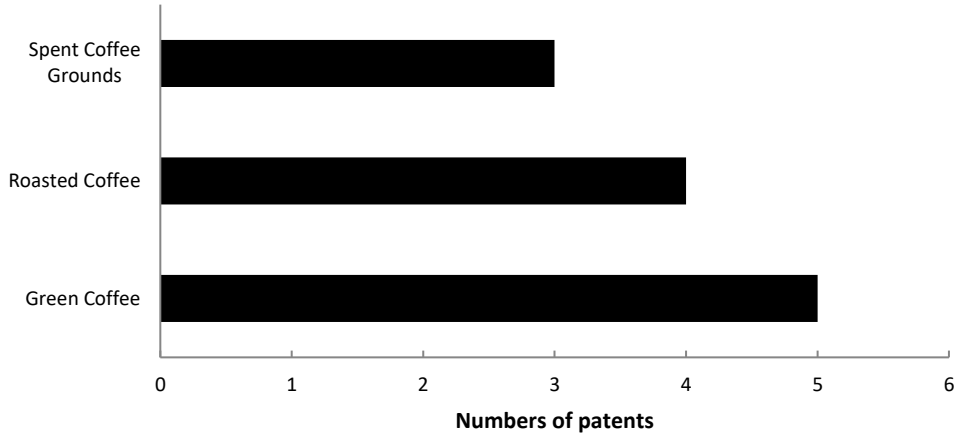

Fig. 7. Raw materials used to obtain coffee oil, according to patents identified at the INPI database

Amongst the patents analyzed on the INPI platform, the highlights were the production of instant coffee with the concentration of aromas from roasted coffee oil, used in pharmaceutical composition for drug absorption and also applied as a stabilizing agent for polymer matrices. Among the patents analyzed data the most cited techniques for the coffee oil extraction were also evaluated (Figure 8).

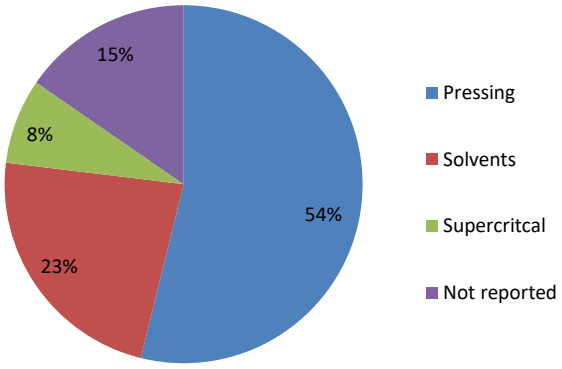

Fig. 8. Coffee oil extraction methods identified at the INPI database

Regarding the technological scope to coffee oil extraction, the pressing, solvent and supercritical fluid was identified. The pressing was the most used technique (54 % of the patents analyzed) to obtain coffee oil. Press extraction is a cleaner technology, without the use of solvents. However, the roasted coffee bean has a high amount of lignin, which makes it difficult to oil recover by pressing [29]. The solvent extraction represented 23 % of the patents analyzed and, among the most used solvents, hexane and ethanol stand out. Regarding the yield, the use of solvents is more satisfactory, justified by the direct interaction between the solvent and lipids inside the vegetal cell. Thus, studies to evaluate new techniques for the breakdown of lignin are necessary and its can allow to the oil recover from toasted coffee more attractive on a large scale.

According to Figure 9, the years 2014 and 2016 presented the highest number of patents filed, considering 1992 as the starting point for first patent filing registration at the INPI, by the company Société des Products Nestlé. The patent PI 9204135-3 A2 deals with the production of instant coffee powder, with the addition of oil to be reintroduced into instant coffee powder as an aroma enhancer [30]. The increase in interest on the part of applicants showed an increase in the number of patent filings on this subject, going from just 3, until the year 2012, to 12 until July 2019, 2014 being the year with the highest number of patents. The patent documents for the years 2018 and 2019 were not reported, due to the confidentiality period, but the number of patents for these years might still increase.

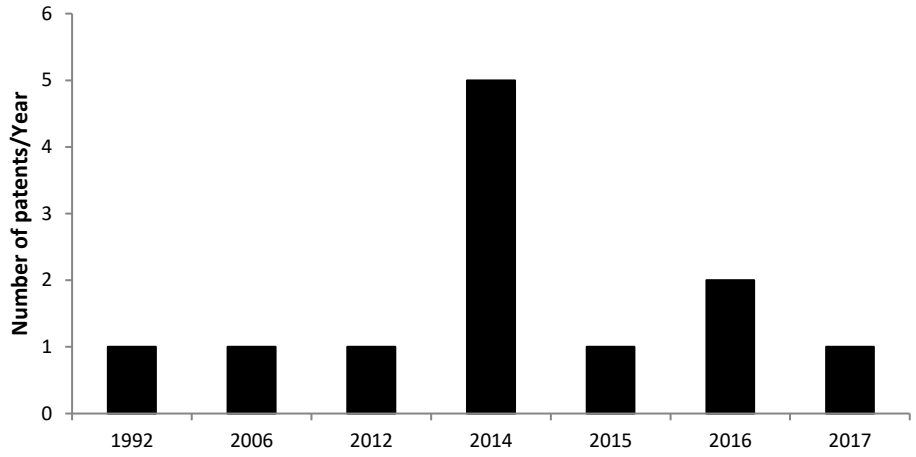

Fig. 9. Temporary evolution of the patent application between 1992-2017 on the INPI basis

Figure 10 illustrates the main countries with patent applications in Brazilian territory, in which it can be seen that Brazil holds 66 % of the patents deposited with the INPI. Among the patents of Brazilian origin, 50 % belong to the Cooperative of Coffee Growers of Gaxupé-MG, while the other half belongs to federal universities such as the Federal University of Goiás, State University of Campinas and Federal University of Pernambuco (UFPE, acronym in Portuguese).

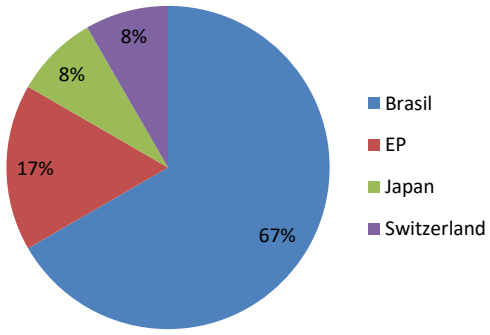

Fig. 10. Numbers of patent per Countries in INPI base

Amongst the universities, UFPE stands out with two patents filed with the INPI. In these cases, one apply the oil extracted from coffee grounds as a raw material for pharmaceutical purposes and the other as a stabilizing agent for polymeric matrices. The patent titled “Use of coffee grounds oil and pharmaceutical composition - BR 10 2016 028713 8” [31] does not describe the oil extraction process, but the use of ground seed oil as a promoter for cutaneous absorption of lipophilic and hydrophilic drugs. The patent - BR 102012024569-8 [32] refers to the use of oil extracted from coffee grounds, as a stabilizing additive of polymer matrices exposed to ionizing radiation. Viscosimeter tests showed a protection of approximately 67 % of the polymeric matrix. Among the main applicants, it was possible to enumerate the percentage of patents that were applied by companies, cooperatives and universities (Figure 11). In this scenario, only legal entities were identified as applicants. In the search any patents were identified when using the combination of the keywords “coffee oil and microencapsulation” and “coffee oil and spray drying”.

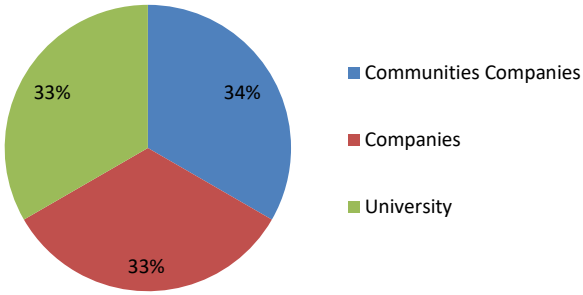

Fig. 11. Kind of Applicants

From these data, it appears that the interest on the part of agencies and universities that promote research addressing the methods and application of coffee oil is still low. Coffee oil is used as a natural additive in foods (antioxidant properties, flavoring) and has potential for application in several industrial sectors. In its entirety, the patents presented in this work focus on using coffee oil directly in food formulations. The extraction of bioactive compounds present in the oil represents an area of technology that needs more attention from researchers. Given the interest of society in seeking new natural compounds and clean technologies for the use of residual biomass aiming to obtain ingredients of industrial interest, studies of this nature have high potential and are still little explored.

4 CONCLUSIONS

This work aimed to present the scenario of patents in the area of extraction and main applications of coffee oil on the platforms available for consultation from INPI and Derwent Innovations Index. In the Derwent database, considering the applied keywords, resulted in 62 selected documents, in which 80 % of the patents were classified as A23 - inserted in the area of food products and their processing; coffee, teas and their manufacture; physical treatment and conservation. Considering only the class A23, 70 % of the documents analyzed within this classification belong to A23F. Within the A23F classification, 21% of the documents analyzed belong to the code A23F-005/46 - coffee aroma; coffee oil; flavoring of coffee or coffee extract (synthetic coffee flavorings). The period between 2014 and 2016 was responsible for 31 % of the evaluated documents and the United States has an expressive leadership with 27.9 % of the patents filed. Only two Brazilian patents were listed in the Derwent Index Innovation database.

Among the major reported technologies for the coffee oil extraction, the supercritical CO_2 represents 70 % of the patents deposited. Among the technological applications of coffee oil stands out the use as an aroma enhancer in the food industry. Nestec SA, a subsidiary of NESTLÉ, was the main depositor, with 14.5 % of the patents. Other private sector companies also stood out such as GENERAL FOOD CORPORATION, Ajinomoto, NESTLÉ and Kraft Food Corporation. In the patents analyzed on the INPI platform, the coffee oil is related to the production of instant coffee with the concentration of aromas, use in pharmaceutical formulation for the drugs absorption and as stabilizing agent for polymer matrices.

Among the most reported extraction methods, pressing represented 54 % of the 12 patents found on the INPI platform. Brazil stood out as the main depositor country, being the main actors in the process linked to cooperatives and universities. The main class of international classification code was C11B (producing, e.g. by pressing raw materials or by extracting waste materials, refining or preserving fatty lanolin, fat oils or waxes; essential oils) with 52 % of analyzed patents. The Cooperative of Cafeeicultures of Gaxupé-MG has 30 % of the patents deposited. No patents were found on the INPI platform related to the keywords “microencapsulation and coffee oil” and “coffee oil and spray-drying”.

This fact requires greater attention on the part of research centers, universities and development institutions regarding the development of patentable inventions correlated with the aforementioned theme. This topic proved to be promising in terms of commercial exploitation due to its efficiency in preserving essential compounds in vegetable oils. The national industry and the academic community must be attentive to the coffee sector that influences the Brazilian economy so much, observing opportunities for constant development and evolution, especially in the transformation into technological innovation.

There is a large area very little explored aiming at the commercial application of SCG for oils, bioactive compounds and co-products recovery. In consequence, very few patents developed for the extraction of SCG compounds with possible application in several sectors such as agriculture, cosmetics and food. Its potential future is for functional foods and natural anti-oxidants production and / or as a flavor enhancer. Many other applications in different areas of society are expected to emerge with the use of coffee oil as a raw material, since the purification of the lipid extract to isolate a single fraction of compounds or a specific molecule is an open field that offers new opportunities for scientific studies and patents. It is necessary to enhance the Brazilian scientific capacity in the

dissemination of technological solutions for the disposal of agro-industrial waste, since this sector has a major role for the country's economy. Furthermore, microencapsulation of SCG's oil can provide an increase in the productive potential and incentive to technological innovation in the agro-industry, as well as the valorization of its residues.

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