Mapping of the Brazilian Groups Studying Nanocellulose

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Abstract: The nanocellulose is a material that has gained much attention in the recent years. So, the relevance of Brazil in this field was evaluated concerning the scientific publications in Web of Science. Next, the Brazilian groups were mapped using a bibliometric procedure on these data. Then, more factors were analyzed from them too. They were the sources to extract the nanocellulose in Brazil, the methods to do it, the characterizations to determine its dimensions and the funding agencies of these researches. The results identified 69 Brazilian groups. Besides, the bacterial cellulose was the most common source. While the acid hydrolysis was the most used method. By its turn, the size characterization was mostly by scanning electron microscopy. At last, the most important agencies were the CNPq, the CAPES and the FAPESP. Giving these points, it was possible to suggest the opportunities to develop the nanocellulose research in Brazil.

Keywords: Nanocellulose; bibliometry; scientometrics; scientific publication; web of science

1 Introduction

The cellulose is the most abundant polymer on the Earth. It is attractive to the environmental friendly applications and to the biocompatible ones too [1]. Some living beings can produce it, like the tunicates, the algae, the bacteria or the plants [2-4].

In fact, it is possible to isolate the cellulose to get a material with at least one dimension in the nanoscale that is known as the nanocellulose. There are many approaches to extract it. They are such as the chemical one, the enzymatic one or even the mechanical one [5]. In the same way, the nomenclature in the field is diverse too. It happens because each morphology leads to a specific name. Thus, there are, e.g., the cellulose nanoparticles, nanofibrils, nanofibers, nanostructures, nanocrystals, whiskers, among others [5-8]. The term nanocellulose is the most general in the area, and it embraces all the possible variations. So it was used in this article.

At the same time, it is well known the cellulose in macro and microscopic scale is studied. In this context, the Brazilian resources and research are recognized worldwide [9,10]. Now on the nanoscale, the situation is quite different. By one hand, some industries in Brazil already produce it. Their nanocellulose is, e.g., from the *Eucalyptus* (supplied by Suzano) or from the bacterial cellulose (supplied by Vuelo Pharma/Membracel) [11,12]. But to the best of our knowledge, there is nothing in the literature about the relevance of Brazilian research on nanocellulose.

For this reason, we aimed to map the main Brazilian groups in this area using a bibliometric procedure. Not only that but also to determine which cellulose sources are the most studied in Brazil. As well as the most frequent methods to isolate the nanocellulose and the techniques carried out to characterize its size. Finally, to verify which funding agencies supported these researches.

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2 Experimental

We followed the bibliometric procedure suggested to analyze the publication and patent data about nanocellulose, with some modifications [13-16]. Here, only the scientific publications were searched. The data were collected on 22 Aug 2018 from Web of Science database from 1945 to 2018. First, the Boolean search expression for topic (TS) was:

TS = (("bacterial cellulos*") OR ("cellulos* crystal*") OR ("cellulos* nanocrystal*") OR ("cellulos* whisker*") OR ("cellulos* microcrystal*") OR ("cellulos* nanowhisker*") OR ("nanocrystal* cellulos*") OR ("cellulos* nano-whisker*") OR ("cellulos* nano-crystal*") OR ("nano-crystalcellulos*") OR ("cellulos* micro-crystal*") OR ("cellulos* microfibril*") OR ("microfibril* cellulos*") OR ("cellulos* nanofibril*") OR ("nanofibril* cellulos*") OR ("micro-fibril* cellulos*") OR ("nano-fibril* cellulos*") OR ("cellulos* microfibril*") OR ("cellulos* nano-fibril*") OR ("cellulos* nanofiber*") OR ("nanocellulos*") OR ("cellulos* nanoparticle*") OR ("nano-cellulos*") OR ("nanoparticl* cellulos*") OR ("nanosiz* cellulos*") OR ("cellulos* nanofill*") OR ("nano-siz* cellulos*") OR ("cellulos* nano-fiber*") OR ("cellulos* nanoparticle*") OR ("cellulos* nano-fill*") OR ("nano-particl* cellulos*")).

This search expression comprehended different nomenclatures used to materials classified as nanocellulose [14].

Second, this initial set of publications was filtered by country (Brazil). However, some publications were about cellulose in the micro or macro scale. Therefore, the papers actually about nanocellulose were selected. The average growth rate (AGR) of publication was calculated from 1991 to 2017 using the annual growth rates, Eq. (1). The year of 1991 was chosen because it was when the Brazilian publications started.

$$G_i = \frac{100(N_i - N_{i-1})}{N_{i-1}} \quad (1)$$

In Eq. (1), G_i is the annual growth rate. N_i is the number of publications in the year "i". N_{i-1} is the number of publications in the year "i-1" [13].

Meanwhile, it was possible to build the matrix of co-authors and apply it to the Grouping of Co-Authors Process (GCAP, developed by the Center for Technological Information in Materials-NIT Materiais, UFSCar, Brazil). This procedure was done to identify the major groups in Brazil. They were confirmed checking the institutions websites and Brazilian Lattes database. Further, we could quantify the main sources to extract nanocellulose. Not only them but also the extraction methods, the techniques to characterize its dimensions and the funding agencies. All data presentations were with Origin® software.

Finally, Fig. 1 summarizes the steps followed to analyze the Brazilian publications.

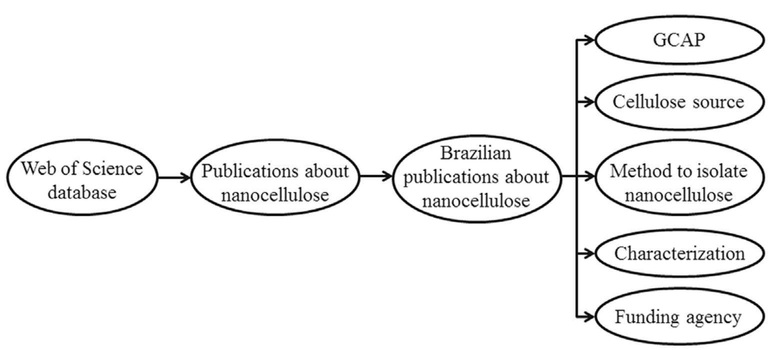


Figure 1: Sequence of steps to analyze the Brazilian publications

3 Results and Discussion

3.1 Brazilian Relevance

The initial set recorded 14,590 publications worldwide from 1945 to 2018. Brazil was related to 645 of them. Fig. 2 presents the global ranking according to Web of Science data considering publications (Fig. 2(a)) and citations (Fig. 2(b)).

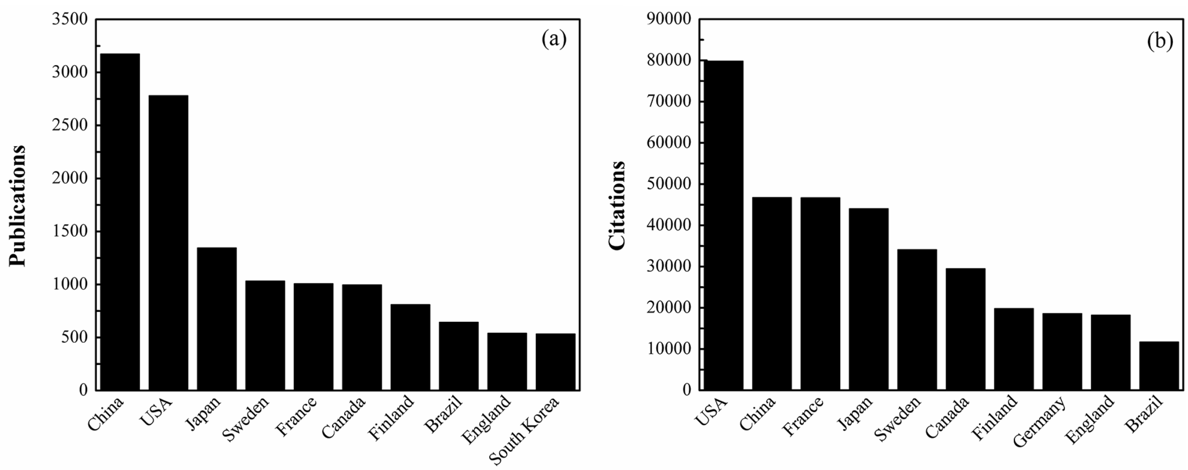


Figure 2: Countries with more publications (a) and more citations (b) about nanocellulose in Web of Science from 1945 to 2018

Brazil was the 8th country with more papers (Fig. 2(a)). It was behind of China, United States, Japan, Sweden, France, Canada and Finland. All these countries were among the most productive ones at publications and patent documents about this theme [7,13]. Brazil overcame even traditional nations as England and South Korea, but it was the 1st in the Latin America production.

However, the world ranking of citations (Fig. 2(b)) is different. Brazil was then the 10th country most cited, behind of Germany and England too. The German position showed in Fig. 2(b) may be explained because of its reviews. There were 33 reviews about nanocellulose written by at least one German author, which showed 7,448 citations. They correspond to 40.0% of citations of German publications. Despite, it is interesting to note that Brazil still is among the most relevant countries globally (Figure 2b) and the 1st in Latin America.

Among the 645 Brazilian papers, only 538 were actually about nanocellulose and written by at least one co-author working in Brazil. Thus, Fig. 3 shows the number of these Brazilian publications per year.

In Fig. 3, it is important to note there are fewer publications in 2018 because the search was realized on August. Almost 8 months of 2018 recorded 73 papers, 78.5% of 2017. It suggests the Brazilian development in this field is still in an emerging stage [13].

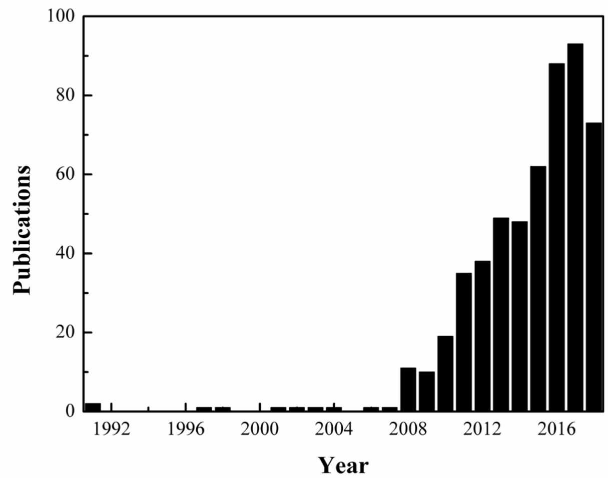


Figure 3: Brazilian publications about nanocellulose in Web of Science from 1991 to 2018

As shown in Fig. 3, the Brazilian studies started to be published in 1991. So, the AGR was from 1991 to 2017, Eq. (1). The world AGR from 1991 to 2017 was 20.9%. It was 20.7% until 2010 [13]. So, the growth of world publications was relatively constant in the recent years. By other hand, the Brazilian publications increased markedly after 2008. The Brazilian AGR from 1991 to 2017 was 54.7%. This justifies why Brazil reached the 8th world position so fast (Fig. 2(a)).

Now considering the language, only 23 papers (4.3%) were in Portuguese. The others (515-95.7%) were written in English. Even most papers published in Brazilian journals were in English (39 of 62). Therefore, this indicates the internationalization of Brazilian science in this area, what has happened in others too [17].

Another relevant fact is the general application of this material. 343 papers (63.8%) at least suggested nanocellulose applied in some nanocomposite, usually as reinforcement. This is a worldwide tendency because these nanocomposites show outstanding properties compared to composites with micrometric cellulose or even with synthetic nanofillers [18]. Around 75% of all nanocellulose publications in the world considered nanocomposites too [8]. They can be used in different areas, such as food packaging materials, biomedical engineering, in the electronics sector and in the oil & gas industry [6,19-21].

3.2 Main Brazilian Groups

Concerning the authorship, 1,475 authors wrote the 538 papers. 1,209 of them (81.9%) were Brazilians or foreigners working in Brazilian institutions. The matrix of co-authors was built and applied to the Grouping of Co-Authors Process (GCAP). There were 69 Brazilian groups. Tab. 1 summarizes the first ten positions with more publications. They wrote 58.3% of all Brazilian production.

Table 1: Main Brazilian groups studying the nanocellulose

Position	Group/Leader	Publications
1	Unesp*-Campus Araraquara / Ribeiro, SJL	57
2	Embrapa#-Unit São Carlos / Mattoso, LHC	39
3	UFMG*/Pereira, FV	32
4	UFSC*/Porto, LM	20
5	Embrapa#-Unit Fortaleza / Rosa, MD	18
6	UFU*/Pasquini, D	17
	UFPR*/Sierakowski, MR	17
7	Unesp*-Campus Araraquara / Guastaldi, AC	16
8	Unesp*-Campus Botucatu / Leao, AL	13
	Uniara§/Trovatti, E	13
9	USP*-Campus São Carlos / Carvalho, AJF	12
	Embrapa#-Unit Colombo / Magalhaes, WLE	12
	UEM*/Rubira, AF	12
	UCS§/Zattera, AJ	12
10	UEL*/Tischer, CA	11
	UFLA*/Tonoli, GHD	11

* Brazilian public universities; # Brazilian public company; § Brazilian private university

According to the data shown in Tab. 1, there are 11 groups of public universities, 2 of private universities and 3 groups of the same public company (Brazilian Agricultural Research Corporation-Embrapa) studying the nanocellulose. The strong presence of public universities is justified because, at least until 2010, over 80% of Brazilian graduate students and researchers were in a public institution [17,22].

It is important to explain about the GCAP that if two authors have at least 50% publications in co-authorship they are considered being from the same research group. For example, prof. H.S. Barud (Unesp) published 54 papers and was the second author with more publications about nanocellulose in Brazil. Over 59% of his papers were with prof. S.J.L. Ribeiro (Unesp), who had 57 publications. Therefore, the GCAP considered they were from the same group (Unesp-Campus Araraquara, leaded by prof. Ribeiro in Tab. 1).

3.3 Main Cellulose Sources

The 538 papers used 66 different cellulose sources. Fig. 4 shows the ten most used. They were present in over 70% of Brazilian papers.

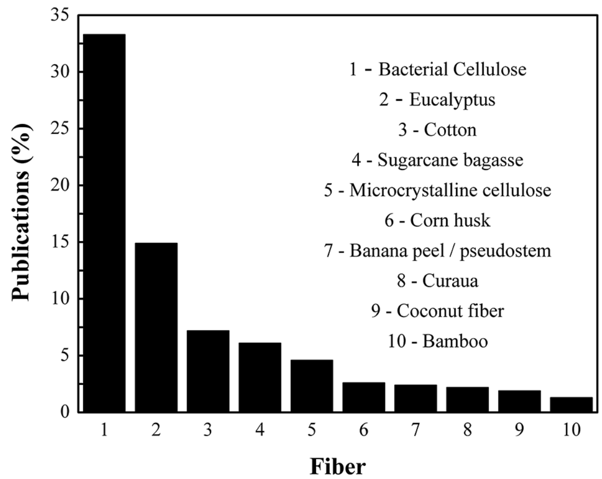


Figure 4: Main cellulose sources in Brazilian publications about nanocellulose in Web of Science

The most common source was the bacterial cellulose (Fig. 4). It happened possibly because the bacterial cellulose is not constituted by pigments, hemicellulose or lignin [3,23,24]. But we must not forget there is always the chance that it occurred simply by a foreigner influence. If the Brazilian scientists had their international experience in groups dedicated to study the bacterial cellulose, then they would be just continuing these researches in Brazil. A future study could analyze the Brazilian international collaboration in this context to confirm it.

Another relevant fact is that 57.5% of Brazilian publications with bacterial cellulose were about biomedical application, such as membranes for bio-regeneration, tissue engineering or drug delivery.

In the meantime, some lignocellulosic residues, as sugarcane bagasse, corn husk and coconut fiber, were among the most used sources (Fig. 4). It must be remembered that Brazil is among the major producers of sugarcane, corn and coconut in the world [25-27]. As a result, lignocellulosic residues from these crops can lead, e.g., to water pollution and microbial deposition [26]. For this reason, to use these residues as a nanocellulose source is an interesting alternative to the environment in Brazil and in others sugarcane producing countries.

As the possible Amazonian sources, there were just 5 of them among the 66. While only curaua (*Ananas erectifolius*) was among the most used (Fig. 4). Its position is explained because its cellulose content is higher than in other lignocellulosic fibers [28,29]. So isolating nanocellulose from curaua is interesting. Besides that, these data reveal the need of more studies using other Brazilian Amazonian sources.

There are at least more two others available cellulose sources: tunicates and algae [2,4,5,30]. Only 2 papers (0.4%) were about nanocellulose from tunicate. Our search found none publication from algae. This presents the opportunities by the lack of studies about animal and algae celluloses.

Most publications (84.8%) studied nanocellulose from only one source. So there is the possibility of more reports comparing several sources as well as their nanocellulose properties.

At last, 10.2% did not specify the cellulose source.

3.4 Main Methods

There are many processes to obtain the nanocelluloses, like the acid hydrolysis, the enzymatic hydrolysis, by ionic liquid or even the mechanical techniques [5,31]. In Fig. 5, there are the most used methods by Brazilian groups.

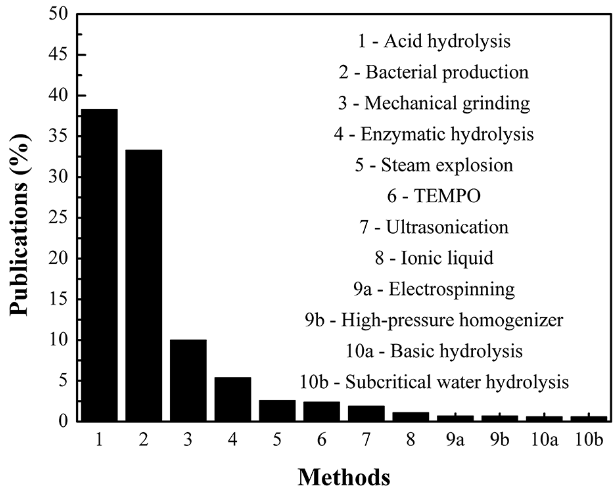


Figure 5: Most used methods to extract nanocellulose in Brazilian publications in Web of Science

The acid hydrolysis is the most common technique worldwide [31]. It happened in Brazil too (38.3%), as shown in Fig. 5. This process usually uses sulfuric acid, at 40-60ºC, for ~45 min [5,18,32]. The crystalline regions of cellulose are resistant to acid attack. However, all the other regions and components are hydrolyzed, allowing to isolate the nanocellulose [31]. The acid hydrolysis is not advantageous because it demands a high energy consumption, to maintain the high temperature during the process. Besides that, it inserts sulfate groups on nanocellulose chains, decreasing its thermal stability, and it generates acid residues [32].

Bacterial production (33.3%) was the second process more used to extract nanocellulose in Brazil, Fig. 5. It means that these publications used the pure bacterial membrane, without an additional treatment. This was expected because this source was the most used by Brazilians (Fig. 4).

Meanwhile, the mechanical grinding was the third method (10.0%). It is one of the most applied mechanical techniques for this purpose in the world too [33]. The cellulose sources are subjected to repeated cyclic stresses and the mechanical energy is the responsible to isolate the nanocellulose [33].

All the other methods in Fig. 5 showed a small contribution in Brazilian papers. This demonstrates the huge need of Brazilian groups studying these techniques in order to obtain more ecofriendly and costless processes.

Unfortunately, 11.0% of the publications did not specify which method was applied to extract their nanocellulose. This value is higher than that which did not identify sources because in some cases, the nanocellulose was purchased or donated by other group or company and the authors only identified the source, not the isolation technique.

3.5 Characterization of Nanocellulose

The measure of nanocellulose dimension is not simple. That happens by its unique shape, its size polydispersity and its propensity for aggregation. Not to mention that to dry it may generate agglomeration. And these new micrometric particles are hard to disperse again [34]. Thus, the choice of the appropriate technique is crucial for the proper characterization [35].

The Brazilian papers about nanocellulose used just four techniques. They were the atomic force microscopy (AFM), the dynamic light scattering (DLS), the scanning electron microscopy (SEM) and the transmission electron microscopy (TEM), Fig. 6(a). Here, SEM was the conventional SEM and the field-emission gun SEM (FEG-SEM) too. In Fig. 6(b), there is a graph with the presence of each technique and of their combinations.

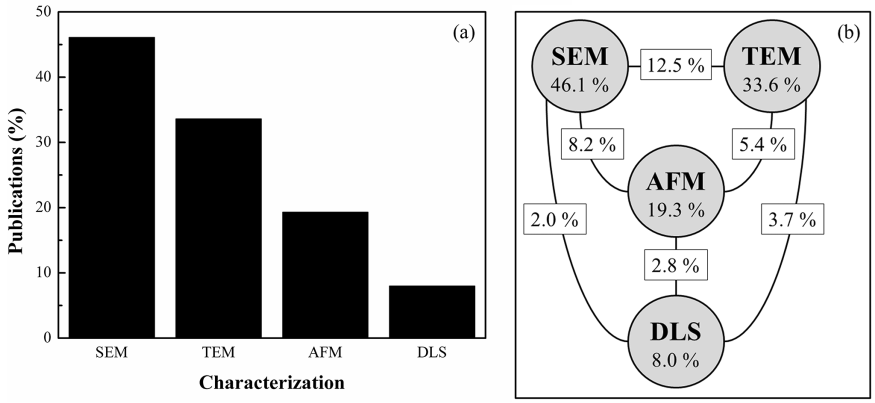


Figure 6: Characterizations used in Brazilian publications about nanocellulose in Web of Science

As shown in Fig. 6(a), SEM was the most used (46.1%). SEM can determine the shape, the length and the width of nanocellulose. However, the height of nanocellulose may difficult the SEM measure. Some particles may be around 5 nm and this can hinder the imaging by the small difference between the heights of the particle and the substrate. In addition, the layer of conductive material (e.g., gold and platinum) usually coating the nanocellulose can change the observed particle size [35].

Going on, the second characterization in Fig. 6(a) was TEM (33.6%). And the combination of SEM and TEM was the most common (12.5%), Fig. 6(b). TEM is usually used to measure the shape, the width and the length of nanocellulose particles [34,35]. It is important to mention that the sample preparation for TEM is the most complex compared to SEM, AFM and DLS. Moreover, lateral association of nanocellulose particles can happen on the TEM's grid, what influence the observed results [34].

The third most used technique was AFM (19.3%), Fig. 6(a). Its results are like TEM measures. But AFM allows analyzing other dimensions, such as the height of nanocellulose [34,35]. Among its limitations there are the possible overestimation of length or height by AFM tip convolution effects and compression of individual particles by the AFM tip too [34].

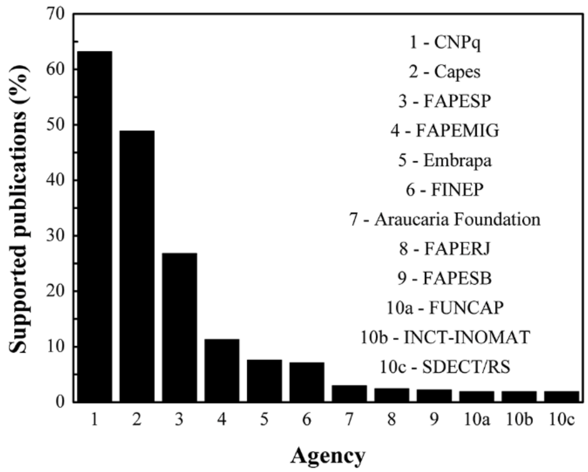
The least used characterization technique showed in the Fig. 6(a) was DLS (8.0%). It gets the hydrodynamic apparent size distribution of nanocellulose. This size is the radius of an equivalent sphere with the same diffusion coefficient. The measure considers all particles (individual ones, aggregates and agglomerates). So, the results cannot be directly related to length or width of nanocellulose [34,35]. However, DLS shows great advantaged too, compared to the others in Fig. 6. For example, the samples can be analyzed just after preparation, without drying on a substrate or grid, and DLS is useful for fast evaluation of particle size, compared to the time-consuming microscopies [34]. Therefore, it is interesting that more Brazilian groups use DLS, at least as a prior step aiming to choose the best condition to a microscopy measure. At least 2.0% of Brazilian publications used DLS as a previous step in characterization, Fig. 6(b). This value could enhance if DLS start to be more used.

A relevant fact is that 28 papers (5.2%) used the facilities of Brazilian Center for Research in Energy and Materials (CNPEM, at Campinas, São Paulo State, Brazil) to the size characterization. The researchers of Brazilian public institutions can use CNPEM facilities for free. They can even receive support for the traveling and the accommodations if they are from outside São Paulo State. But it is important to cite that the groups with no author working at Campinas wrote only 8 publications (1.5%).

Recent studies about the nanocellulose characterization considered other technique. They stated that the field flow fractionation (FFF) may analyze the size distribution of the suspension. It would be more accurate than the results of DLS or microscopic techniques [34,36,37]. However, our search recorded none Brazilian paper that used FFF. This reveals the opportunity for the next years of Brazilian researches using FFF.

3.6 Financial Support

Which are the funding agencies that support the Brazilian researches about nanocellulose? Our search recorded 104 agencies. Among them, 43 were Brazilians and 61 were foreigners. The Fig. 7 summarizes the first ten positions of agencies with more supported publications in this area.



CNPq*: National Council for Scientific and Technological Development
Capes*: Higher Education Improvement Coordination
FAPESP#: São Paulo State Research Foundation
FAPEMIG#: Minas Gerais State Research Foundation
Embrapa*: Brazilian Agricultural Research Corporation
FINEP*: Studies and Projects Funding
Araucaria Foundation#
FAPERJ#: Rio de Janeiro State Research Foundation
FAPESB#: Bahia State Research Foundation
FUNCAP#: Research Support Foundation of Ceará State
INCT-INOMAT*: National Institute of Science and Technology in Functional Complex Materials
SDECT/RS#: Science, Technology and Development Secretariat of Rio Grande do Sul State

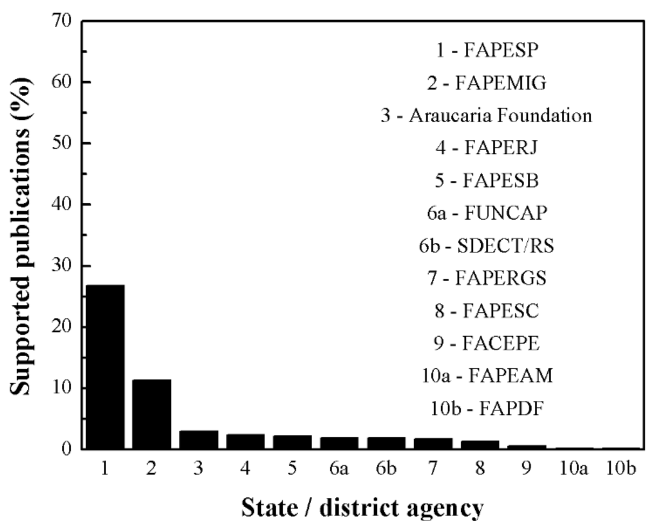
*Federal public agency; #State public agency

Figure 7: Main funding agencies that supported Brazilian publications about nanocellulose in Web of Science

In Fig. 7, there are only Brazilian agencies. They all were public agencies, related to the Federal Government (CNPq, CAPES, Embrapa, FINEP and INCT-INOMAT) or to a State Government (Araucaria Foundation, FAPESP, FAPEMIG, FAPERJ, FAPESB, SDECT-RS and FUNCAP).

Remarkably, CNPq and CAPES supported 200 papers (37.2%). This shows the relevance of these agencies, individually and coupled, to the Brazilian research in this field.

By its turn, in Fig. 8 there is the ranking of all funding agencies related to State or Federal District government that supported Brazilian publications about nanocellulose.



Agency	Brazilian State / District
FAPESP	São Paulo
FAPEMIG	Minas Gerais
Araucaria Foundation	Paraná
FAPERJ	Rio de Janeiro
FAPESB	Bahia
FUNCAP	Ceará
SDECT/RS	Rio Grande do Sul
FAPERGS	Rio Grande do Sul
FAPESC	Santa Catarina
FACEPE	Pernambuco
FAPEAM	Amazonas
FAPDF	Federal District

Figure 8: Main funding agencies related to State or Federal District government that supported Brazilian publications about nanocellulose in Web of Science

There is a relation between the first six State agencies (Fig. 8) and where the main research groups are in Brazil (Tab. 1). FAPESP is from São Paulo State, where Unesp, USP and Uniara are. Meanwhile, FAPEMIG is from Minas Gerais State, where UFMG, UFU and UFLA are. In like manner, Araucaria Foundation is from Paraná State, where UFPR, UEM and UEL are too. At last, SDECT-RS and FAPERGS are from Rio Grande do Sul State, where UCS is. These facts indicate the key role of State agencies to support research as well. It is important to mention that all the research groups in Tab. 1 received financial support from the Federal agencies too.

Analyzing the ranking in the Fig. 8, there are still 16 Brazilian States that supported none research about nanocellulose. This situation could change. Therefore, more groups can be created and grow up across the country, not only in South and Southeast regions.

Last, 82 publications (15.2%) did not specify their financial support.

4 Conclusions

Brazil is among the most important countries in nanocellulose research in the world. It is the first in Latin America too. The main Brazilian groups in this field were mapped, showing the strong presence of public institutions.

Besides, the cellulose sources were analyzed. The data revealed that the Brazilian researchers had used to prefer the bacterial cellulose and the lignocellulosic fibers. In contrast, the nanocellulose from the Amazonian sources, the tunicates and the algae still need to be more studied in Brazil. In like manner, further publications comparing more than one source are necessary too.

Now considering the methods to extract the nanocellulose, the acid hydrolysis was the most used. Then, there were the bacterial production and the mechanical grinding. While the others showed no significant presence. Therefore, it is interesting an enhancement in the use of distinct techniques. Not only that but also how they can be applicable on a large-scale production. Further, the Brazilian publications could consider the economic feasibility of their methods and sources too. In this manner, the innovations in these procedures would be useful for the environment and the whole society. Likewise, they would raise the Brazilian relevance worldwide too.

Going on, the characterization of the nanocellulose size was by SEM, TEM, AFM and DLS. Although they all show limitations, their results are reliable. On the other hand, new techniques have been used too, such as field flow fractionation, but not in Brazil yet. So, the Brazilians could also carry out these new approaches. For example, they may start with an international collaboration and, eventually, purchase the equipment. One institution that is able to perform it, for example, is CNPEM. Moreover, a higher visibility of CNPEM across the country should be encouraged, with the opportunities to use its facilities.

It is important to remember that other physicochemical properties are crucial in this field too. We may mention, e.g., the chemical composition, the crystallinity degree and the thermal stability. Here we analyzed only the nanocellulose size. Because of that, future studies can quantify the main techniques used to characterize each of these properties.

Concerning the funding agencies, CNPq and CAPES supported 63.2% and 48.9% of Brazilian publications, respectively. In addition, 16 States still did not invest in this field yet. The public investment could increase trough these State agencies for example. But we must not forget that the nanocellulose is a key material for many industries, such as the paper, the food packaging and the biomedical. By this reason, the support from the private sector should increase too.

Further suggestions for future researches include to identify the international collaboration of Brazilian groups in this area and to map the groups in other prolific countries.

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