

Cellulose-Based Hydrogels: Patent Analysis

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Abstract: *Background:* Among biopolymers, cellulose and its derivatives are the most commonly used for hydrogel formulations. The innovation and improvement of cellulose-based hydrogels concerned the raw materials, synthesis and methods of preparation, formulations and fabrication processes, as well as applications.

Objectives: This study, in the form of patent analysis, presents the state by introducing what has been innovated and patented concerning cellulose-based hydrogels.

Methods: Three databases have been used in this study: the Patentscope, the Espacenet, and the Lens patent data set. A detailed analysis has been provided regarding publication dates, patent families, jurisdictions, inventors, applicants, owners, and patent classifications.

Results: A total of 8053 patent documents related to cellulose-based hydrogels have been published between 1965 and 2021. The United States leads the patent race in this sector, and the Massachusetts Institute of Technology is one of the top academic applicants.

Conclusion: Based on patent classifications, most patent documents are related to medicinal preparations characterized by special physical forms. More specifically, the classification concerns materials for prostheses or coating prostheses, including cellulose derivatives characterized by their function or physical properties, such as macromolecular gels, hydrogels, or hydrocolloids.

Keywords: Cellulose, hydrogel, formulation, innovation, patent family, intellectual property.

1. INTRODUCTION

Over the past two decades, the use of polymeric materials has increased significantly for biomedical applications. Each of the applications requires materials with specific physical, chemical, biological, biomechanical, and other properties to give effective treatment. Polymeric biomaterials have several advantages over metallic or ceramic materials, including ease of processing, low cost, and availability with desired mechanical and physical properties, as well as the ability to fabricate them in a variety of forms (e.g., latex, film, sheet, fibers, hydrogels, etc.) [1].

Polymers in the form of hydrogels are considered functional and intelligent polymers. Hydrogels are defined as crosslinked hydrophilic viscoelastic structures that are capable of swelling in water and/or biofluids and forming insoluble three-dimensional (3D) molecular networks. The network remains at equilibrium in an aqueous medium due to the balance of elastic forces of the crosslinked polymer and osmotic forces of the environmental liquid [2].

The 3D structure of hydrogels is due to crosslinking that forms an insoluble structure in the biofluid

environment. The elasticity and mechanical properties of these structures, as well as the presence of a large amount of water, offer a resemblance to various human biological tissues, which allows them to be used in many biomedical applications. For example, in tissue engineering applications, the reticulated 3D structures of hydrogels enable the maintenance and support of cells. Furthermore, hydrogel materials are used in this area because they closely mimic biological tissue and can support the growth, multiplication, and differentiation of cells to produce regenerated organ transplants [3].

Hydrogels can be made from a variety of water-soluble materials including synthetic polymers (e.g., poly(lactide-co-glycolide), poly(ethylene glycol), poly(vinyl alcohol), etc.), and others, as well as natural polymers such as protein-based polymers (e.g., fibrine, silk, collagen, etc.), and polysaccharide-based polymers (e.g., alginate, chitosan, cellulose, etc.) [4].

Cellulose is the most commonly and abundantly synthesized polymer in nature. It belongs to the polysaccharides that constitute the major fraction of the plant wall. It is a linear homopolymer made up of many (1→4)-linked β -D-glucopyranose units [5]. Cellulose and its derivatives are the most commonly used biopolymers for hydrogel formulations. They exhibit rheological and biologically appropriate properties that

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make them suitable for biomedical applications [6-8]. Hydroxypropylmethylcellulose and carboxymethylcellulose are cellulose derivatives that belong to the large family of cellulose ethers and are widely used in the field of tissue engineering. As hydrogels, these two cellulose derivatives are used as synthetic matrixes for cartilage regeneration [9,10] and bone tissue regeneration [11,12].

The first patent application concerning cellulose-based hydrogels was filed in 1963 and then granted in 1965 [13]. Through this patent, the inventors have proposed methods of producing pellets of a hydrogel containing carboxymethylcellulose. In particular, the cellulose-based hydrogel has been fabricated by adding drops of an aqueous solution of a water-soluble salt of carboxymethylcellulose (2% w/w) to an aqueous solution of an aluminum salt (e.g., aluminum nitrate, aluminum sulfate, ammonium aluminum chloride, etc.) [13].

Research on cellulose and its derivatives as hydrogels is developing rapidly through the innovation and improvement of raw materials, chemical synthesis, and methods of preparation and formulation. Moreover, research in this field has been actively growing for the past couple of decades. This is also evident from the increase in the number of patent applications filed each year worldwide in this area [14].

This study, in the form of patent analysis, presents the state by introducing what has been innovated and patented concerning cellulose-based hydrogels between 1965 and 2021. Furthermore, a detailed analysis is provided regarding publication dates, patent families, jurisdictions, inventors, applicants, owners, and patent classifications. Finally, an overview, which gives an analysis of the past, present, and future trends, is proposed by selecting five relevant patents in this area.

2. MATERIALS AND METHODS

2.1. Resources

The supported field codes used in this study were based on the Patentscope search service of the World Intellectual Property Organization (WIPO) [14,15], the Espacenet of the European Patent Office (EPO) [16], and the Lens patent data set [17].

2.2. Methods

During the search, different keywords and related terms to cellulose-based hydrogels were used, and

patents were searched according to title, abstract, and claims. The search was then filtered to include only documents with a publication date until December 31st, 2021. Then, a detailed patent analysis has been provided regarding publication date, patent families, jurisdictions, inventors, applicants, owners, and patent classifications.

3. RESULTS AND DISCUSSION

3.1. Patent Documents

8053 patent documents were found after the search. Generally, it encompasses patent applications and granted patents. About cellulose-based hydrogels, the found patent documents are classified as 5885 patent applications and 2168 granted patents. Hereinafter, the state of the art will be reviewed by introducing what has been patented concerning cellulose-based hydrogels. A detailed analysis of patents for the used cellulose and its derivatives, extracting methods/processes, chemical synthesis, formulations, and applications will be provided following these sections: publication dates, patent families, jurisdictions, inventors, applicants, owners, and patent classifications.

3.2. Publication Dates

A patent publication is a step when the patent document (e.g., patent application, granted patent, etc.) is made available to the public, to which a publication number and a publication date have been assigned by a patent authority. In other words, the date on which a patent document is published, thereby making it part of the state-of-the-art [18].

Concerning cellulose-based hydrogels, the evolution of patent documents as a function of the published date is illustrated in Figure 1. Between January 1st, 1965, and December 31st, 2021, a total of 8053 patent documents concerning cellulose-based hydrogels were found, with an average of 143 patent documents per year. The starting year of patenting, 1965, recorded only one granted patent. However, the year 2020 recorded the maximum number of 684 patent documents, as well as the maximum number of 534 patent applications and 150 granted patents. Furthermore, last year, cellulose-based hydrogels were the subject of 592 patent documents, with 485 and 107 patent applications and granted patents, respectively.

3.3. Patent Families

A patent family is a collection of interrelated patent applications filed in one or more countries (or regions)

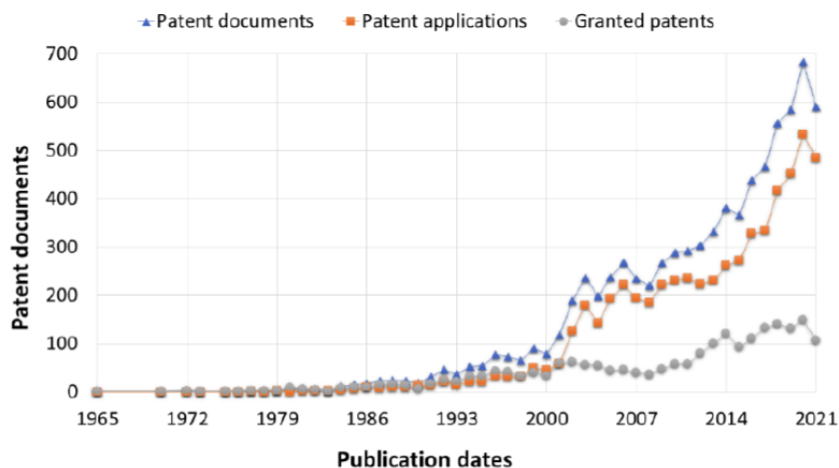


Figure 1: Evolution of patent documents (patent applications and granted patents) for cellulose-based hydrogels as a function of the published date.

to protect the same or similar invention by a common inventor and linked by a common priority (or priorities). In other words, that means several different patent documents whose technical content is considered identical. Two family types are considered: the simple family and the extended family. The first one is a collection of patent applications covering the same technical content. However, the second one is a collection of patent applications covering similar technical content [18,19].

For cellulose-based hydrogels, a total of 8053 patent documents were found, with 4274 simple families and 3972 extended families. These results mean that 4274 patent documents cover the same technical content as 3972 patent documents that cover similar technical content but are published at different times in the same country or published in different countries or regions.

3.4. Jurisdictions

An applicant, or a first mentioned applicant in the case of joint applicants, can apply for a patent at the appropriate patent office (e.g., United States Patent and Trademark Office (USPTO), Moroccan Office of Industrial and Commercial Property (OMPIC), China National Intellectual Property Administration (CNIPA), etc.) under whose jurisdiction he normally resides, has his domicile, has a place of business, or the place from where the invention originated [20]. Under such regional systems, an applicant requests protection for an invention in one or more member states of the regional organization in question. The regional office (e.g., African Intellectual Property Organization (OAPI), European Patent Office (EPO), Eurasian Patent

Organization (EAPO), etc.) accepts these patent applications, which have the same effect as national applications, or grants patents if all the criteria for the grant of such a regional patent are met [21].

About cellulose-based hydrogels, the top 10 jurisdictions of filled patents between 1965 and 2021 are illustrated in Figure 2. The United States through the USPTO encompasses 3741 patent documents, with a higher patent contribution per total of 46.77%. On the other hand, the global system for filing patent applications, known as the Patent Cooperation Treaty (PCT) and administered by WIPO, encompasses 1970 patent documents with a patent contribution per total of 24.63%. Thirdly, the EPO, through which patent applications are filed regionally (in Europe), encompasses 1084 patent documents with a patent contribution per total of 13.55%. Finally, China, through the CNIPA, encompasses 849 patent documents with a lower patent contribution per total of 10.62%.

3.5. Inventors

An inventor is a natural person designated for a patent application. In several cases, the inventor can also be the applicant, and there may be more than one inventor per patent application [18].

Concerning cellulose-based hydrogels, the top 10 inventors are illustrated in Figure 3. Nuopponen Markus is ranked as the first inventor who has recorded 60 patent documents. As for the podium in second place, it is shared between the two inventors, Bennett Steven and Laukkanen Antti, who have recorded 47 patent documents each. In third place, inventor Yliperttula Marjo has recorded 40 patent documents. It

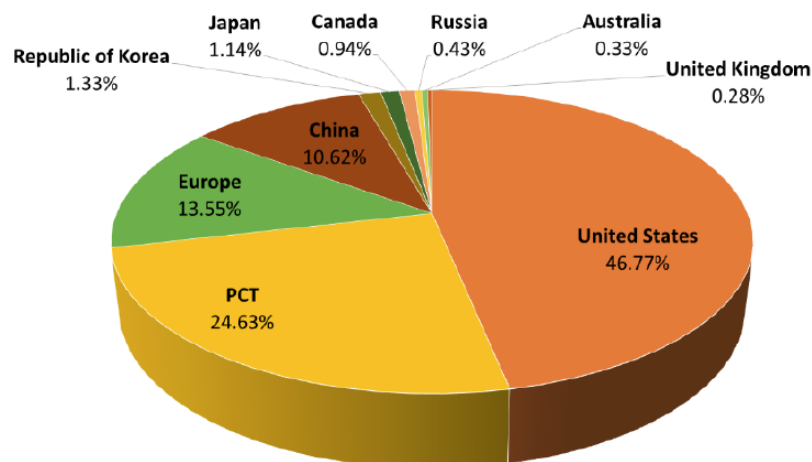


Figure 2: Jurisdictions (top 10) of resultant patent documents for cellulose-based hydrogels.

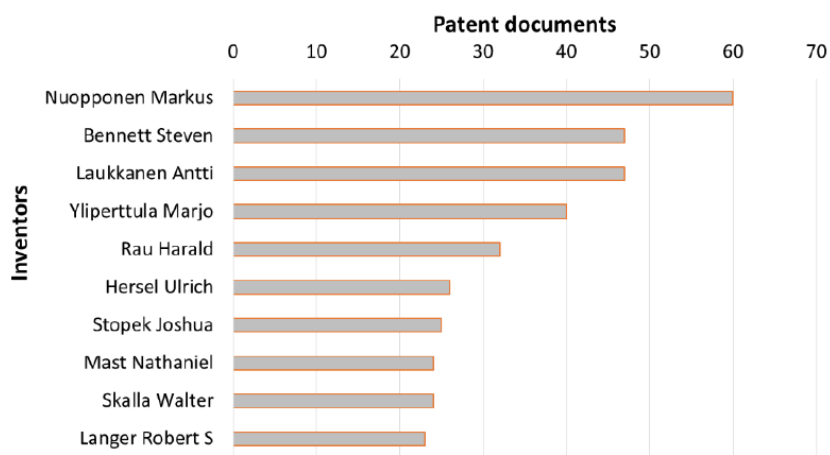


Figure 3: Inventors (top 10) of resultant patent documents for cellulose-based hydrogels.

should be noted that the patent documents of the three inventors, Nuopponen Markus, Laukkanen Antti, and Yliperttula Marjo, are primarily concerned with the company Upm Kymmene Corporation (Helsinki, Finland).

3.6. Applicants

An applicant is a person (i.e., a natural person) or an organization (i.e., a legal entity) that has filed a patent application. In several cases, the applicant can also be the inventor, and there may be more than one applicant per patent application [18].

Concerning cellulose-based hydrogels, the top 10 applicants are illustrated in Figure 4. As a legal entity, Upm Kymmene Corporation (Helsinki, Finland) is ranked as the first applicant that has recorded 127 patent documents. In the second place, the applicant, Procter & Gamble (Cincinnati, OH, United States), as a legal entity, has recorded 119 patent documents.

Thirdly, the legal entity Alza Corporation (Mountain View, CA, United States) has recorded 81 patent documents. Finally, it should be noted that among the top 10 applicants are three academic institutions. Massachusetts Institute of Technology (Cambridge, MA, United States), University of California (Oakland, CA, United States), and Harvard College (Cambridge, MA, United States) have recorded 61, 55, and 48 patent documents, respectively.

3.7. Owners

An assignee, or patent owner, is a person (i.e., a natural person) or an organization (i.e., a legal entity) to whom the inventor or applicant assigned the right to a patent. The patent owner has the right, for a period limited to the duration of the patent term, to protect his brainchild. The patent system stops others from making, using, or selling the invention without the inventor's permission or requires others to use the invention under agreed terms with the inventor [18].

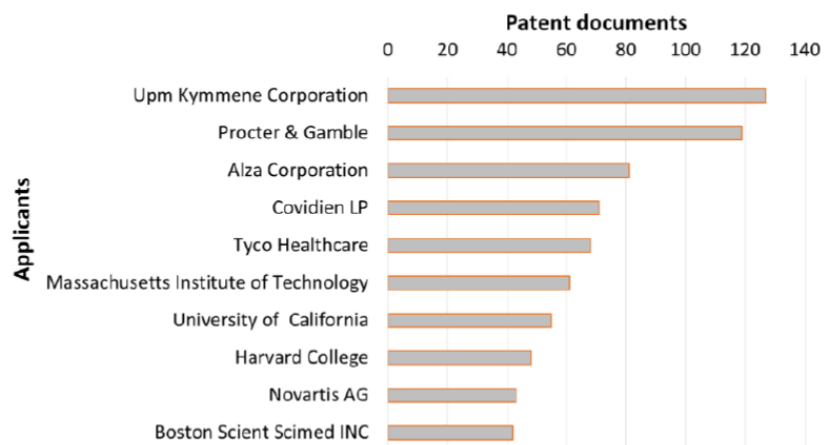


Figure 4: Applicants (top 10) of resultant patent documents for cellulose-based hydrogels.

Concerning cellulose-based hydrogels, the top 10 owners are illustrated in Figure 5. As a legal entity, Covidien LP (Mansfield, MA, United States) is ranked as the first owner who has recorded 151 patent documents. In the second place, the owner, Upm Kymmene Corporation (Helsinki, Finland), as a legal entity, has recorded 49 patent documents. In third place, the owner, Novartis AG (Basel, Switzerland), as a legal entity, has recorded 44 patent documents.

3.8. Patent Classifications

The International Patent Classification (IPC) is a hierarchical system in the form of codes, which divides all technology areas into a range of sections, classes, subclasses, groups, and subgroups. It is an international classification system that provides standard information to categorize inventions and evaluate their technological uniqueness [22].

Concerning cellulose-based hydrogels, the top 10 IPC codes are illustrated in Figure 6. The most

common IPC code corresponds to A61K9/00 which is a group of medicinal preparations characterized by a special physical form. This group recorded it alone with 955 patent documents. Secondly, the subgroup C08J3/075 which represents macromolecular gels is presented within 591 patent documents. Thirdly, the subgroup of cellulose derivatives (A61K47/38) is presented within 579 patent documents. For more information about the top 10 IPC codes, a description of each IPC code is shown in Table 1.

4. INNOVATION ON CELLULOSE-BASED HYDROGELS

The formulation of hydrogels is a technological innovation in the field of tissue engineering. These hydrogels are developing rapidly through the improvement of the raw polymers, formulations, preparation methods, as well as fabrication processes. Described herein are the most relevant patents focused on cellulose-based hydrogels in the cases of formulation and preparation methods (Table 2).

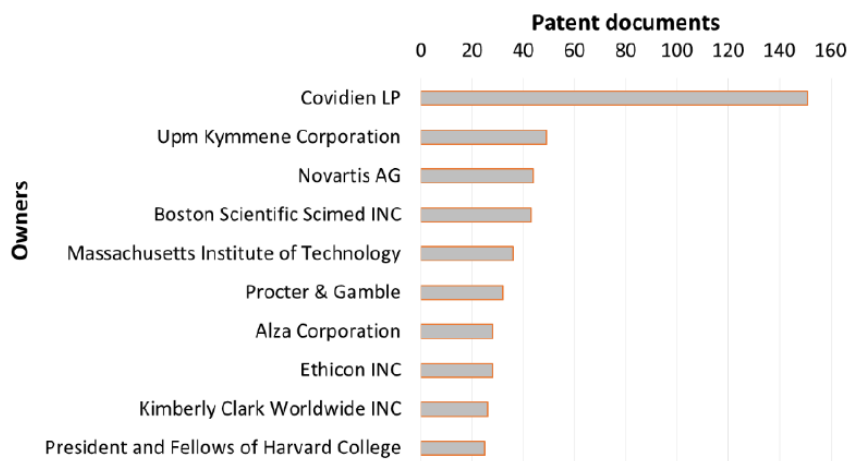


Figure 5: Owners (top 10) of resultant patent documents for cellulose-based hydrogels.

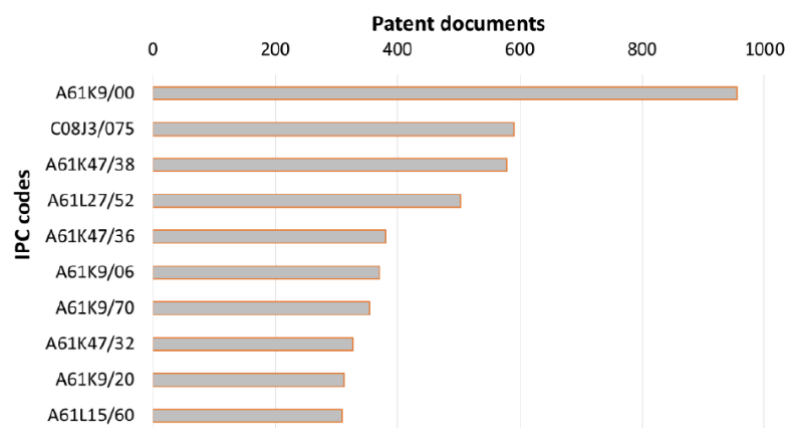


Figure 6: IPC codes (top 10) of resultant patent documents for cellulose-based hydrogels.

Table 1: Meaning of IPC Codes (Top 10) in Relation to Patent Documents for Cellulose-Based Hydrogels [22]

IPC	Description
A61K9/00	Medicinal preparations characterized by special physical form
C08J3/075	Macromolecular gels
A61K47/38	Cellulose derivatives
A61L27/52	Materials characterized by their function or physical properties: Hydrogels or hydrocolloids
A61K47/36	Polysaccharide derivatives
A61K9/06	Medicinal preparations characterized by special physical form: Ointments
A61K9/70	Medicinal preparations characterized by special physical form: Web, sheet, or filament bases
A61K47/32	Macromolecular compounds obtained by reactions only involving carbon-to-carbon unsaturated bonds (e.g., carbomers)
A61K9/20	Medicinal preparations characterized by special physical form: Pills, lozenges, or tablets
A61L15/60	Liquid-swellable gel-forming materials (e.g., super-absorbents)

Table 2 provides the information from the relevant patent documents to demonstrate the innovation and improvement of cellulose-based hydrogels. The selection of these patent documents is based on the type of patent documents (i.e., patent applications [24,27], and granted patents [23,25,26]), the most prolific countries patenting on cellulose-based

hydrogels (i.e., the United States [25,26] and China [27]), as well as the patenting levels (i.e., USPTO [25,26], CNIPA [27], EPO [23], and WIPO [24]).

In 2002, Saga and Saito invented a formulation concerning a transparent cellulose hydrogel. The invention is related to a transparent cellulose hydrogel

Table 2: Details of Selected Relevant Patents in the Area of Cellulose-Based Hydrogels

Patent number	EP0872275B1	WO2011079380A1	US8871016B2	US10307722B2	CN110804192A
Patent document	Granted patent	Patent application	Granted patent	Granted patent	Patent application
Title	Transparent cellulose hydrogel and process thereof	Transparent bacterial cellulose nanocomposite hydrogels	Cellulose-based hydrogels and methods of making thereof	Method for reducing the viscosity of a nanofibrillar cellulose hydrogel	Cellulose antibacterial hydrogel and preparation method thereof
Simple Families	6	4	2	7	2
Extended Families	8	4	10	7	2
Priority date	April 17, 1997	December 30, 2009	August 3, 2011	December 22, 2014	September 29, 2019
Filed date	April 17, 1998	December 23, 2010	August 3, 2011	December 21, 2015	September 29, 2019
Published date	March 20, 2002	July 7, 2011	October 28, 2014	June 4, 2019	February 18, 2020
Reference	[23]	[24]	[25]	[26]	[27]

useful as a raw material for an ophthalmic material such as a soft contact lens, an artificial crystal lens, an artificial cornea, or an artificial vitreous body. The hydrogel is comprised of cellulose having a hydroxyl group that is not chemically crosslinked. It is formed by physical crosslinking via hydrogen bonding between the hydroxyl groups in cellulose. To prove the concept, the inventors have proposed an aqueous cellulose solution by dissolving sodium cellulose xanthate in an aqueous alkali solution. Then, they proposed other organic solvents as alternatives, such as dimethyl sulfoxide/paraformaldehyde, dimethylformamide/nitrogen tetroxide, and dimethylformamide/chloral. Based on the handling of the solution and the mechanical strength of the gel, the inventors have affirmed in some embodiments that the cellulose source should preferably show a polymerization degree of approximately 200–1000, a concentration of approximately 5–10% w/w, and a viscosity of approximately $100\text{--}2.10^5$ cP at 20°C [23].

In 2011, Li *et al.* invented a transparent polymeric nanocomposite hydrogel. The invention is related to a polymeric nanocomposite hydrogel made from a water-insoluble polymer (i.e., poly(2-hydroxyethyl methacrylate)) and a water-insoluble nanofiber (i.e., bacterial cellulose). In the patent application, the inventors described and claimed polymeric nanocomposites that are produced through free radical polymerization of hydroxyethyl methacrylate monomer in the presence of bacterial cellulose with the assistance of ultrasound to enhance the mixing of bacterial cellulose, initiator, and the monomers. The polymeric nanocomposite hydrogel is then formed by the immersion of the dry polymeric nanocomposite in water. As a result, the transparent polymeric nanocomposite hydrogel and compositions presented a high transmittance, a preferred bacterial cellulose loading of less than 0.1%, water content of about 40% in weight, and good mechanical integrity and strength. Finally, the inventors have recommended polymer nanocomposite hydrogels and compositions for hydrogel applications, particularly contact lenses and optical components for biosensors [24].

In 2014, Trexler *et al.* invented cellulose hydrogels with different physicochemical properties, such as high-water content, high transparency, high permeability, high biocompatibility, high tensile strength, and optimal thickness. In the granted patent, the inventors described and claimed a process for preparing a cellulose hydrogel that comprised a solvent to activate the cellulose, the removal of the solvent from the

activated cellulose, the formation of a cellulose-based solution, the gelation of the cellulose-based solution, and optionally the drying and rehydrating of the gel. The inventors have affirmed in some embodiments that the cellulose hydrogel is a composite comprised of different celluloses (i.e., microcrystalline cellulose and bacterial cellulose). Furthermore, the wet cellulose hydrogel has been obtained with the following properties: a cellulose content of approximately 2–9% w/w, a tensile strength of approximately 50–600 kPa, a tear strength of approximately 0.10–0.60 N/mm, a strain to failure of approximately 40–80%, a suture retention strength of approximately 0.05–0.30 N/mm, transparency that exceeds 85% at 550 nm, Young's modulus of approximately 100–700 kPa, and puncture resistance of approximately 50–300 kPa. As an application, the inventors have finally proposed cellulose hydrogels for use as contact lenses [25].

In 2019, Nuopponen *et al.* invented a method for reducing the viscosity of a nanofibrillar cellulose hydrogel. The invention is related to a dispersion comprised of a nanofibrillar cellulose hydrogel and an aqueous growth medium containing salts and sugars for cell culture. In the granted patent, the inventors described and claimed a nanofibrillar cellulose hydrogel comprised of a concentration of nanofibrillar cellulose of approximately 0.05–4% w/w, an aqueous growth medium comprised of a concentration of NaCl of approximately 140–160 mmol/l and a concentration of D-glucose of approximately 5–55 mmol/l. The inventors have affirmed in some embodiments that the nanofibrillar cellulose may be anionic nanofibrillar cellulose, and the nanofibrillar cellulose hydrogel and the aqueous growth medium may be mixed at a volume ratio in the range of about 9:1–1:9, or 3:1–1:1. Finally, the inventors have proposed the nanofibrillar cellulose hydrogel for maintaining or culturing cells, and the cellulose nanofibers could be degraded enzymatically by adding enzyme mixtures, such as cellulase-hemicellulase [26].

In 2020, Zhong *et al.* invented a preparation method for a novel biocompatible and antibacterial hydrogel based on bacterial cellulose. The invention is related to a biomedical gel with the advantages of rapid gelling reaction, a simple reaction system, and high gel plasticity. In the patent application, the inventors described and claimed a preparation method for bacterial cellulose/Fmoc-L-phenylalanine antibacterial hydrogel by adopting an in-situ chemical crosslinking process. The inventors have affirmed in some embodiments that the antibacterial hydrogel prepared

by rapid gelling reaction and a simple reaction system has high mechanical strength, broad-spectrum antibacterial effect, and good biocompatibility. To prove the concept, the inventors have proposed different formulations comprised of a concentration of Fmoc-L-phenylalanine of 6 mg/ml and a concentration of bacterial cellulose of approximately 2–8 mg/ml. Furthermore, results confirmed that formulations have a specifically good antibacterial effect on staphylococcus aureus and bacillus subtilis, as well as good biocompatibility, biodegradability, no toxicity, and high mechanical strength [27].

5. CONCLUSION

In recent years, cellulose and its derivatives have been extensively used as hydrogels for tissue engineering and other applications. For this study, the term “cellulose” includes not only the cellulose biopolymer but also the derivatives associated with it. These derivatives in the form of polysaccharides can be of any type, including cellulose ethers (e.g., methyl cellulose, carboxymethyl cellulose, etc.) and cellulose esters (e.g., cellulose acetate, cellulose butyrate, cellulose nitrate, etc.) originating from straw, wood, cotton, or bacteria.

This study concerned patent analysis concerning cellulose-based hydrogels. More specifically, it concerned innovation and improvement between 1965 and 2021. A detailed analysis has been provided regarding publication dates, patent families, jurisdictions, inventors, applicants, owners, and patent classifications. During a search, a total of 8053 patent documents (5885 patent applications and 2168 granted patents) were found for cellulose-based hydrogels, and 2020 was the year with the maximum number of patent documents (684). The United States leads the patent race in this sector with 3741 patent documents, and the Massachusetts Institute of Technology is one of the top academic applicants.

Based on the patent classification codes, all filled patents and most inventions are intended for medicinal preparations characterized by special physical form, as well as materials characterized by their function or physical properties. The knowledge clusters and expert driving factors of this patent analysis indicate that the research and development based on the formulation of cellulose-based hydrogels are concentrated in most patents. More specifically, the research and development outline concerns: (i) cellulose derivatives; (ii) macromolecular gels; and (iii) hydrogels or hydrocolloids.

Finally, the selected relevant patents in the area of cellulose-based hydrogels have been discussed. The synthesis of inventions through these patents showed that major claims concerned raw materials, polymer chemical synthesis, methods of preparation, formulations, and fabrication processes, as well as applications.

LIST OF ABBREVIATIONS

3D	=	Three-dimensional
CNIPA	=	China National Intellectual Property Administration
EAPO	=	Eurasian Patent Organization
EPO	=	European Patent Office
IPC	=	International Patent Classification
OAPI	=	African Intellectual Property Organization
OMPIC	=	Moroccan Office of Industrial and Commercial Property
PCT	=	Patent Cooperation Treaty
USPTO	=	United States Patent and Trademark Office
WIPO	=	World Intellectual Property Organization

AVAILABILITY OF DATA AND MATERIALS

The data presented in this study is available within this article's content.

FUNDING

This research received no external funding.

CONFLICT OF INTEREST

The author declares that this article's content has no conflict of interest. The author has no relevant affiliations or financial involvement with any organization or entity with a financial interest in or financial conflict with the subject matter or materials discussed in this article.

ACKNOWLEDGEMENTS

The author acknowledges the WIPO for the Patentscope search service, the EPO for the Espacenet patent search, and the Cambia Institute for the Lens patent data set used in this study.

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Received on 28-05-2022

Accepted on 21-07-2022

Published on 25-08-2022

<https://doi.org/10.6000/1929-5995.2022.11.03>

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