



## Bibliometric mapping of cellulose nanoparticle research (2013–2023) using VOSviewer

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### Abstract

Cellulose is the most abundant biomaterial widely regarded as the most abundant organic substance and polysaccharide. Various materials, including cotton, ramie, algae, bamboo and tunicate, can be used as raw materials for nanocellulose. This study aims to do a mapping analysis using the VOSviewer application. The keyword "cellulose nanocrystal" is utilized in a scientific database using Scopus. Based on a search of 7,752 relevant publications published between 2013 and 2023. The bibliometric analysis was conducted using the VOSviewer software and Scopus Analytics. VOSviewer provides two separate visualization methods: network and overlay. 89,2% of journal articles, 6,3% of review papers, and 4,5 % of conference papers were obtained. Cellulose nanocrystals (CNC) and the isolation method of nanocrystals have been essential research subjects. China is the leading country in publications and research collaborations with cellulose nanocrystal subjects. An increasing trend until 2023 in cellulose nanocrystal research is the International Journal of Biological Macromolecules and Polymers. The best resource is Materials Science. Various isolation methods have been used to isolate CNC from various raw materials.

*Keywords: cellulose nano crystal, Bibliometric analysis and Scientific mapping*

### 1. Introduction

Cellulose nanocrystals can be extracted mainly from plants, algae, empty palm oil bunches, etc. Cellulose is considered the most abundant biopolymer on earth, and nanocellulose material can decompose naturally within a few weeks, with good biodegradability. Besides having good biodegradability, cellulose is a renewable material, so its production can be sustainable. Applications in the biomedical field, cellulose nanocrystals can be made into aerogels, reinforcing materials in composites, low-calorie carbohydrate substitutes in food, absorbents, oil emulsifiers, coating agents, food, electronics, the development of electronic and automotive materials is one form of utilization of nanocrystals [1]. Cellulose generally has high strength, low density, large surface area, and excellent biodegradation capabilities, so it has great potential for development [2]. Cellulose, which is known as anhydroglucose cellulose in its linear form, also comes from  $\beta$ 1-4 bonded glucopyranose units. Hydrogen bonds can unite oxygen and hydroxyl atoms and form cellulose chains [3]. Cellulose can be derived from

cellulose-abundant substances in diverse configurations, such as fibers, microfibers, nanofibers, and nanocrystals [4]. CNC has advantages, including high thermal stability, good mechanical strength, low cytotoxicity, a large surface area, and natural properties that are 100% environmentally friendly[5][6]. Cellulose nanocrystals are nanoscale cellulose that can be produced in diverse morphological configurations, including round, cylindrical, flat, or elongated [7]. Although many techniques have been employed to create CNCs, the procedures typically involve three steps: First, pretreatment; subsequent, bleaching; and last, acid hydrolysis [6].

Cellulose is a natural biopolymer that can be applied in various fields, including the paper, textile, food, pharmaceutical and medical industries. CNC is applied in the food packaging industry because it produces a transparent film and has the advantage of being less sensitive to humidity because its crystallinity is higher than CNF. The disadvantage of higher crystallinity is that the resulting film is brittle.

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Receive Date: 27 December 2023, Revise Date: 14 February 2024, Accept Date: 01 April 2024

DOI: 10.21608/ejchem.2024.258587.9095

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So, it is necessary to add additives to improve the mechanical properties of the resulting product. Natural additives such as sorbitol should be added to maintain oxygen impermeability up to 70% relative humidity (RH). Pure Carboxymethyl cellulose (CMC) and CNC-CMC composites can extend the shelf life of food products [8]. CNC applications are found in many fields, including medicine, health, pharmaceuticals, cosmetics, environmental food, materials and electronics.

This research required bibliometric analysis of several globally published papers on nanocrystalline cellulose. The objective is to present a comprehensive summary of global output and identify the current and future directions of research. Bibliometric analysis is a systematic evaluation technique that employs statistical analysis, data mining, and mathematical methods to reveal emerging trends in a particular academic subject. It is becoming increasingly essential and is utilized in several study fields. Bibliometric analysis is a quantitative research method that utilizes data mining, mathematics, and statistics to detect emerging trends within a particular academic discipline. Presently, it is becoming more and more widespread and employed in several academic fields [9]. The VOSViewer software, designed to generate and display bibliometric maps, analyzed the global publication landscape. Readers can construct and view a network or connection using a text-mining feature while citing an article or topic. It can display and navigate through longer articles and publications utilizing a variety of options and functions, such as zooming, searching and scrolling. VOSViewer can exhibit and represent specific information in the Bibliometrics graphic map [10].

## 2. Experimental

Bibliometric analyses incorporate mathematical and statistical methods to assess bibliographic data [11]. The bibliometric review approach aims to understand the connections between journal citations and research success, offering a short overview of the current status of a thriving or emerging research field. Bibliometric analyses extract the necessary data for a bibliometric investigation from many citation databases, such as Scopus and Web of Science. The study utilizes research data extracted from publications published in Scopus-indexed journals. The keyword "cellulose nanocrystal" searches for published or published data according to the title, keywords, and abstract criteria. Thus, 7,752 articles were assessed according to the chosen topic. The articles taken are articles published in 2013-2023. The collected articles are then saved in \*.ris format. \*.ris files are bibliographic references for written literature. To export database search results to

reference managers such as Mendeley, RefWorks, ProCite, Zotero, or Covidence, one frequently uses the RIS format. Furthermore, the data is visualized and analyzed as a bibliometric map. Data from the database source that has been prepared is then mapped in two forms, namely network visualization and density visualization [12][13].

## 3. Results And Discussion

### 3.1 Bibliographic extraction

A search string containing the keyword "cellulose nanocrystal" was typed into the search field on the Scopus website (<https://www.scopus.com/>).

Table 1. explains that the bibliographic investigation yielded results of 7752 research documents, as shown. Articles and reviews from 2013 to 2023 were all included in this collection of materials. All research output was indexed in the Scopus database. There are various applications of cellulose nanocrystals in medicine, food, and materials. Various applications utilize the inherent scale of activities to develop cellulose nanocrystals from various materials, isolation methods, and applications. The proliferation of research publications in this field can be attributed to the increasing fascination it has garnered in both academic and professional spheres.

**Tabel. 1.** Bibliographic extraction

Type of documents	2013-2023	
	Number	percentage
Article	6917	89,2
Review	488	6,3
Conference paper	347	4,5

Fig. 1 shows documents per year by source from Scopus. Analyzing search results can be seen in the increasing trend in a publication by the International Journal of Biological Macromolecules and Polymers, which is proof that research on nanocrystalline cellulose is still exciting and can be developed further. Fig. 2, the document that discusses these keywords the most, is related to materials science from Fig. 3. China has emerged as the foremost nation in terms of publications and research collaborations pertaining to cellulose nanocrystals, with an impressive total of about 2500 articles published between 2013 and 2023.

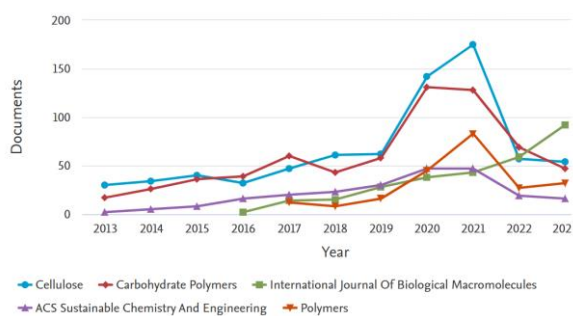


Fig. 1. Documents per year by source from scopus Analyze search results.

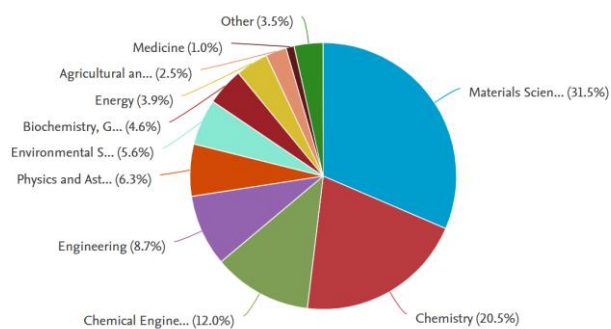


Fig. 2. Document by subject area from scopus Analyze search results.

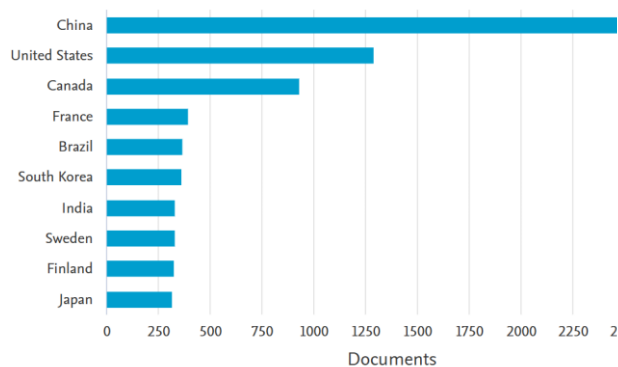


Fig. 3. document by country.

### 3.2 Overview of research performance

#### 3.2.1 Visualization of cellulose nanocrystal topic areas using VOSviewer

VOSviewer is used to map the acquired article data computationally. The quality of the outcomes from bibliometric mapping can be enhanced by network analysis. In network analysis, standard methods include visualization, clustering, and metric evaluation.

Specific publication themes and keywords are associated with each other according to co-occurrences, co-authorship, and place of origin [11]. Additionally, color is used to show how relevant and

commonly used a specific study is. The more a phrase is utilized in multiple research disciplines, the more starkly its related line contrasts. Conversely, if the hue is bright, a weak association is suggested [9].

The network maps generated by VOSviewer utilize a two-dimensional map that represents the relationships between items based on their distance. Greater distances and vice versa suggest stronger and more resilient correlations. A shorter distance, conversely, indicates a more robust connection. The size of the circle represents the term's significance. The computational mapping yielded 68 items. Every phrase linked to cellulose nanocrystals in the data mapping was separated into five clusters. This cluster has been divided according to a collection of related terms. Each cluster is divided as follows:

**Cluster 1** has 27 items marked in green, namely acid hydrolysis, acidolysis, acids, bacterial nanocellulose, bacterium, bagasse, bleaching, carbon nanotube, carboxymethyl cellulose, carboxymethylcellulose, cellulose, crystal, curcumin, enzymatic hydrolysis, enzyme activity, hemicellulose, hydrochloric acid, hydrolysis, lignin, micro fibrillated cellulose, nano-cellulose-nanocellulose, sodium hydroxide, sulfuric acid, sulfuric acid hydrolysis.

The keyword "acid hydrolysis" represented this cluster with 295 occurrences. Acid hydrolysis is the most common method for isolating cellulose nanocrystals and carboxymethyl cellulose. Besides breaking down the cellulose microfibrils, it converts some of their surface hydroxyl groups into esters [14]. Cellulose extraction techniques through chemical processes include pretreatment, alkalization, acid treatment, oxidative bleaching, and mechanical processes (sonication, homogenization). CNC production consists of three main parts: fiber pretreatment, CNC isolation, and hydrolyzed cellulose post-treatment. Fiber pretreatment includes alkali treatment and bleaching treatment. Post-processing of hydrolyzed cellulose includes purification and sonication [15].

Pretreatment is essential in removing lignin and large amounts of hemicellulose from the biomass. Cellulose polymers have amorphous and crystalline structures. Amorphous material is easily accessible in polar solutions. Alkalization converts crystalline cellulose to alkaline cellulose by breaking the hydrogen bonds in the inter-crystalline structure of the cellulose [16].

Cellulose nanocrystals (CNC) are generally isolated in four stages: depolymerization or alkylation process, bleaching, acid hydrolysis, and mechanical dispersion [17]. In the Alkali Process, cellulose fibers are contacted with a robust alkaline solution such as KOH or NaOH to remove substances that dissolve in alkali. To expose some of the substances that dissolve in alkali to remove several hemicelluloses and other impurities covering the outer surface of the fiber cell

walls. Alkaline treatment aims to disrupt the OH bonds in the fiber network structure by ionizing the hydroxyl groups to become alkoxides. Alkalization of cellulosic materials causes an increase in crystallinity because much of the hemicellulose is removed. The alkaline process adds costs to the process and creates environmental problems. Replacing the strong base with water has been done by adding cellulose treated with electron beam irradiation. This method is effective in reducing lignin and hemicellulose content [18].

Another keyword that is important in CNC is bleaching after delignification with oxygen in an elemental chlorine-free (ECF) procedure; chlorine dioxide is often used to perform bleaching. The leftover lignin is preferentially oxidized by chlorine dioxide, which finally makes it soluble. Some chlorinated phenolics do, however, form. It is possible to bleach without using chlorine dioxide by applying oxygen (O), ozone (Z), and peroxide (P) in that order in a fully chlorine-free (TCF) procedure [19]. Bleaching/bleaching after alkaline treatment means there is still lignin content in the fiber, which needs to be reduced, including by the bleaching process. Bleaching includes using  $\text{NaClO}_2$  and  $\text{H}_2\text{O}_2$  as precursors. When using sodium chlorite, alkaline bonds will form between lignin and hemicellulose after alkali treatment, inhibiting lignin removal. However, chlorine is not environmentally friendly, so other alternatives are needed. Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) can break bonds and act as an oxidizer to remove lignin. The steps are carried out until white tissue is obtained [20]. The isolation process aims to break down the amorphous part to extract the crystalline part from cellulose.

*Acid hydrolysis* is a method that can be widely used to produce CNC by removing amorphous groups to produce cellulose nanocrystallites with high crystallinity. Acid hydrolysis forms half-sulfate ester groups on the surface of CNC, which causes the formation of high colloidal stability for CNC dispersion in water due to electrostatic repulsion. CNC has a rod shape with a length of 50-350 nm with a diameter of 5-10 nm. Highly crystalline CNC can be applied to films, coatings, reinforcements in composite materials, or as additives [21]. Initial treatment, type of solvent, extraction method, bleaching method, length of time, temperature, and other treatments influence the characteristics and yield of the CNC produced.

Temperature and time in research show that variations in time affect the yield produced, where extraction using acid hydrolysis with an extraction time of 90 minutes produces a yield 25% greater than 22 minutes for 60 minutes [22]. Acid hydrolysis isolation commonly used sulfuric acid ( $\text{H}_2\text{SO}_4$ ), phosphoric acid ( $\text{H}_3\text{PO}_4$ ), hydrochloric acid (HCl),

nitric acid ( $\text{HNO}_3$ ), and a mixture of mineral and organic acids  $\text{H}_2\text{SO}_4$  has been proven to be effective in removing the amorphous component of cellulose fibers and producing stable CNC suspensions.

Acid hydrolysis using sulfuric acid is most commonly used, with 375 occurrences. The thermal stability of CNCs prepared with sulfuric acid can be improved by neutralizing the CNCs (22). CNCs isolated from lignocellulosic biomass rely mostly on sulfuric or hydrochloric acid hydrolysis. However, it has disadvantages, including corrosion, excessive cellulose degradation, environmental pollution, and use of large amounts of water [23].

Enzymatic hydrolysis is related to 157 occurrences. Enzymatic hydrolysis is a relatively easy method. Using highly corrosive reagents and large amounts of water is unnecessary to obtain CNC. In addition, there is less energy consumption and low water volume. However, the yield obtained is generally lower than the purification process using physicochemical methods [24]. CNC production using an enzymatic process is an environmentally friendly method, does not produce acid residue, and can maintain the original properties of cellulose, such as structure and thermal stability. However, there is a difficulty, namely that it takes a long time. The research showed that CNC production was carried out with hydrolysis times of 48 and 72 hours. 0.35 mg dry mass of cellulose produces approximately  $1.6 \times 10^{11}$  CNC/mL [25]. The high cost required for the enzyme isolation method, long processing time, and relatively low yields are still weaknesses of enzymatic hydrolysis technology [26].

Also, in this cluster, there are 6311 occurrences with the keyword cellulose, proving how cellulose is very interesting to research and develop as a sustainable raw material. Bacteria are used in the manufacture of bacterial nanocellulose (BNC) with 50 occurrences. *Acetobacterxylinum* is one of the bacteria most frequently used to make BNC (*Gluconacetobacterxylinus*). BNC can be produced by the microbes *Achromobacter*, *Agrobacterium*, *Acetobacter*, *Acanthamoeba*, *Alcaligenes*, *Pseudomonas*, *Rhizobium*, and *Sarcina*, as well as by the algal species *Rhizoclonium*, *Cladophora*, *Chaetomorpha*, and *Microdictyon* [27][28]. Carboxymethylcellulose, which has 92 occurrences, or carboxymethylcellulose, is one such polymer. Because of its pH-dependent swelling properties, it is utilized as a drug delivery method in hydrogel form [29] [19].

Carboxymethyl cellulose with 92 occurrences. Carboxymethyl cellulose is a linear, long-chain, water-soluble anionic polysaccharide. Carboxymethylation and oxidation are methods of creating carboxyl groups on the surface of cellulose nanofibrils. The initial carboxymethylation treatment on cellulose fibers is carried out by replacing the

hydroxyl groups with carboxymethyl groups in an alkaline medium. Carboxymethyl groups produce fewer passes during mechanical fibrillation due to increased electrostatic repulsion between fibrils. The ease in the fibrillation process is caused by reduced hydrogen bond strength and hydrophobic properties in cellulose fibers [30]. Carboxymethylation, oxidation, and sulfonation are some ways of adding ionic groups to the surface of cellulose fibers. This process makes the surface negatively charged, forming a more stable suspension of carboxymethylated fibers and enhancing the breakdown of lignocellulosic fibers down to nanoscale size. Nanocellulose is nano-sized cellulose (1-100 nanometers) [31].

Carboxymethyl cellulose (CMC) is a cellulose derivative of a water-soluble anionic polysaccharide with high molecular weight obtained by treating cellulose with chloroacetic acid. CMC can hold water and has good water absorption properties because the hydroxyl groups found in CMC can form water bonds through hydrogen bonds—the weaknesses of CMC, especially its very hydrophilic nature and poor thermomechanical properties. Carboxymethylation can be used as a pretreatment in the CNC manufacturing process. The carboxymethylation process is carried out by adding monochloroacetic acid [30].

Carboxymethylation in nanofibers breaks the hydrogen bonds between cellulose molecules, causing damage to the nanofiber structure and a decrease in the physical properties of the resulting product [32]. Carboxymethylation can be used as a pretreatment in making cellulose nanocrystals. During carboxymethylation pretreatment, the C<sub>6</sub>, C<sub>2</sub>, and C<sub>3</sub> positions on the chain structure of cellulose. Carboxymethylation causes a nonselective reaction, increasing carboxyl group content, reducing interfibril adhesion, and facilitating microfibril separation [33]. Disadvantages of carboxymethylation include excessive depolymerization and the presence of negatively charged surfaces. However, the significant reduction in energy used during the manufacture of carboxymethyl cellulose is one of the advantages of using this treatment [34].

**Cluster 2** has 27 items marked in blue, namely 2,2,6,6 tetramethylpiperidi, alginate, alginates, amides, amines, ammonium persulfate, carboxylation carboxylic acid, carboxylic acids, cation, crystalline cellulose, crystal, enzymes, flow kinetics, hydroxy group, kinetics, nanocrystal, nanofiber, nanoparticle, nanoparticles, oxidation-reduction reaction, oxidation-reduction, oxidized cellulose, sodium alginate, surface functionalization, 2,2,6,6-Tetramethylpiperidine-1-oxyl (TEMPO), TEMPO-mediated oxidation, surface functionalization, TEMPO, TEMPO mediated oxidation.

TEMPO in the second cluster with 167 occurrences. TEMPO oxidation to enhance the surface

charge of CNCs. The utilization of TEMPO oxidation facilitates the production of consistent and clear aqueous suspensions of nanocelluloses while preserving their initial shape [35]. The weakness of isolation with TEMPO is that it is expensive, so its use is limited. Ammonium persulfate (APS) can be an option to replace TEMPO. APS is a simpler process, that produces CNCs-COOH compounds. This process can directly remove lignin, hemicellulose, and other impurities simultaneously. The limitations of the APS method are the amount of APS required for biomass conversion (22.8 g APS per g biomass) and the long reaction time (minimum 16 hours). The use of tetramethylethylenediamine (TMEDA), in combination with ultrasonic assistance and other methods, is expected to overcome the weaknesses of APS [36].

In this cluster, APS has 50 occurrences. Cellulose can be derived from algae. Further modification and functionalization of the -1,4-glycosidically connected anhydroglucose units are made possible by the chemical makeup of the cellulose molecule, namely the presence of three reactive hydroxyl groups on each of these units. Hydroxyls become oxidized functions, and one of the key elements in influencing the macroscopic characteristics and chemical behavior of cellulosic materials is the presence of carbonyl (mostly aldehydes, CHO, and ketones, CO) and carboxyl (COOH) groups [37]. The main reaction that occurs between chlorine dioxide and carboxylic acids is with the reducing end groups of carbohydrates [38].

Alginate with 122 occurrences. Alginate and cellulose nanocrystals are most often combined in applications in the fields of medicine and food. Alginate served as the network polymer for the production of hydrogels [39]. The mechanical characteristics of the composite films, including alginate, were lower than those of typical polymer films, restricting their ability to provide additional functionalities. Cellulose nanocrystals are frequently employed as a reinforcement in packaging to enhance the tensile strength, as previous reports have verified. Cellulose nanocrystals can serve as a load-bearing element. When the polymer chains transfer their payload to the nanocrystals, it results in an enhancement of the tensile strength [40].

**Cluster 3** has seven items marked in yellow, namely cellulose derivatives, cellulose nanocrystals, cellulose nanocrystals (cncs), cellulose nanocrystal (cncs), chitin, nanocomposite, nanocrystals. Cellulose is a derivative in the third cluster with 4007 occurrences. The cellulose content in raw materials is one of the important parts of carrying out CNC isolation. Cellulose nanocrystals (CNCs) were obtained from the extracted cellulose [41]. Cellulose derivatives, including methylcellulose, ethylcellulose, hydroxypropyl cellulose, and carboxymethyl

cellulose, are widely employed in film production due to their favorable characteristics.

**Cluster 4** has four items: cellulose nanoparticles, crystalline structure, microcrystalline cellulose, and nanocrystalline cellulose. The keyword "micro-crystalline cellulose represented this cluster with 103 occurrences and crystalline structure with 74 occurrences. The crystalline structure of cellulose nanocrystals was characterized using X-ray diffraction (XRD) spectroscopy. XRD for studying structural characteristics of cellulose nanocrystal. By carrying out XRD analysis, the transformation from amorphous to crystalline can be seen by Comparing the XRD pattern of the sample. XRD is also used to measure the degree of crystallinity[42]. The key component influencing cellulose's crystallinity is the crystalline region. The range of crystallinity is 60-80%, contingent upon the different cellulose sources [43]. thermo-gravimetric analysis (TGA) is used for the analysis of thermal degradation in CNC. CNC-reinforced nanocomposites may be subjected to high temperatures and thermal deterioration during melt processing, which may impair the nanocomposites' mechanical and other functional qualities. Thus, it is crucial to comprehend thermal behavior and, in turn, the stability of these CNC nanocomposites [44]. A crucial element in the development of novel formulations for the production of new composites is thermal stability. At 310 °C, cellulose nanofibrils with a 5–40 nm diameter begin to decompose and continue until 400 °C [45]. Transmission electron microscopy (TEM). TEM is used to measure the characterizations of the shape and size. CNCs contain short rod-like or whisker-shaped structures with diameters ranging from 2 to 20 nm and lengths ranging from 100 to 500 nm [46]. Scanning electron microscopy (SEM). SEM can be used in the characterization of nanomaterials, detailing the surface information and the morphologies of CNC [47]. Cellulose nanocrystals (CNCs), sometimes referred to as nano-whiskers, are hard, rod-shaped, or spherical particles that are a few hundred (100–1000) nm long and 30 nm or less in width[48]. Dynamic light scattering (DLS) technique. DLS technique to measure rod-like cellulose nanocrystal (CNC) particle length (L) also estimates the average length of CNC [49]. Fourier-transformed infrared (FTIR) spectroscopy identifies the various functional groups and determines the chemical components[50]. One thing that influences the characteristics, shape, particle size, and crystallinity of the CNC is the CNC isolation method. It is very important to determine the isolation method [51].

**Cluster 5** has three items marked in purple, namely carbon dioxide, cellulose nanocrystals (CNC), and cellulose nanocrystals (CNC). In this cluster, cellulose nanocrystal (CNC) had 103 occurrences. Food packaging uses CNC due to its ability to generate

a transparent film and its superior resistance to humidity, which is attributed to its higher crystallinity than CNF. The disadvantage of higher crystallinity is that the resulting film is brittle. So, it is necessary to add additives to improve the mechanical properties of the resulting product. Additives in food packaging, such as sorbitol, are added to maintain oxygen impermeability up to 70% relative humidity (RH).

Carboxymethyl Cellulose (CMC) and CNC-CMC composites can extend the shelf life of food products. CMC has been employed as a material with high oxygen and carbon dioxide barrier properties. CMC is utilized as a food ingredient to confer stability and viscosity to foods, owing to its elevated viscosity at relatively low concentrations. Due to its biodegradability and biocompatibility, it has been extensively utilized in the preparation of biodegradable films among cellulose derivatives [8]. CNC applications are found in many fields, including medicine, health, pharmaceuticals, cosmetics, environmental food, materials, and electronics. Network visualization aims to visualize whether the network or relationship between research terms is strong or not, as can be seen in Fig.4.

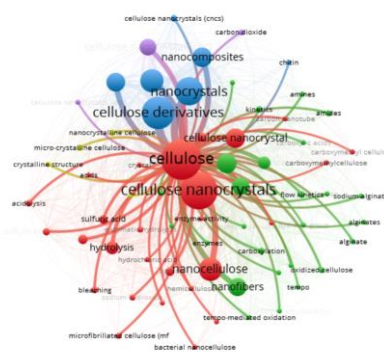


Fig. 4. Network visualization

Density functions are to display density or emphasis on the research group. The density displayed in Fig 5 shows that most research is related to cellulose nanocrystals, cellulose nanocrystal derivatives, and isolation using hydrolysis, and acid hydrolysis is the most common method with a bright yellow colour. The brighter the colour, the more research there is. The research is still relatively limited for domain carboxymethyl and carboxyl cellulose nanocrystal and other isolation methods besides acid hydrolysis, marked with an off-colour. In this way, there are opportunities for renewable research by taking these items.



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