Abstract: Polysaccharides are formed by a long chain of monosaccharides, with the main function of promoting energetic and structural reserves for plants and animals. They can be applied as a base of electrolytes, using ionic liquids (ILs) as a solvent base. The study of electrolytes is an emerging field, as they are applied as secondary batteries, fuel cells, solar cells, supercapacitors and chemical sensors. They operate stably under extreme conditions, maintaining their high thermal stability. Furthermore, their low cost and environmentally safe character, compared to conventional electrolytes, have attracted considerable attention in the scientific field. ILs are composed entirely of ions and could be potentially applied as solvents. As electrolytes, ILs are environmentally friendly, and their use in combination with polysaccharides leads to a synergic effect. In the present study, a systematic review was performed of all papers published from 2014 to 2022 regarding ILs and polysaccharides through a search of three databases. Due to the large number of results found, only papers about electrolytes were considered and the main findings described. This study allows for easy identification of the most relevant fields of study with respect to ILs and polysaccharides, as well as the main gaps to be explored in the literature.

Keywords: electrolytes; ionic liquids; solvent media; systematic review

1. Introduction

Polysaccharides are formed by a long chain of monosaccharides, with the main function of promoting energetic reserves and structural integrity for plants and animals. All polysaccharides are linked by α-glycosidic or β-glycosidic bonds, depending on the type of polysaccharide. Starch and glycogen have glucose bonds linked by α-glycosidic bonds [1–3]. As shown in Figure 1, the main difference between cellulose and glycogen or starch is the higher amount of hydrogen bonds between adjacent glucose units. These bonds exist both within a chain and between adjacent chains, resulting in tougher fibers than those of glycogen or starch.

Polysaccharides from natural sources have been used for various applications, such as to promote anticancer [4] and antitumor activity [5], for immunomodulatory dietary applications [6], adsorption of petroleum [7], reinforcement of composites [8–11] and production of nanoreinforcements [12,13], among others. On the other hand, ionic liquids (ILs) possess inherent characteristics, such as low cost and environmentally safe characteristics.
(depending their chemical configuration (cation and anion selection)), compared to conventional electrolytes; therefore, they have attracted considerable attention in the scientific field [14–17].

|          | Cellulose | Starch   | Amylose | Amylopectin | Glycogen |
|----------|-----------|----------|---------|-------------|----------|
| Source   | Plant     | Plant    | Plant   | Animal      |          |
| Subunit  | β-glucose | α-glucose | α-glucose | α-glucose   |          |
| Bonds    | 1–4       | 1–4      | 1–4 and 1–6 | 1–4 and 1–6 |          |
| Branches | No        | No       | Yes (~per 20 subunits) | Yes (~per 10 subunits) |          |
| Diagram  |          |          | [Cellulose Diagram] | [Starch Diagram] | [Amylose Diagram] |
| Shape    |           |          | [Cellulose Shape] | [Starch Shape] | [Amylose Shape] |

Figure 1. Characteristics of the main polysaccharides (adapted from: https://medicinalherbals.net/polysaccharides (accessed on 11 January 2021)).

Among their several advantages, ILs can operate stably under extreme conditions, maintaining their high thermal stability. Furthermore, ILs are composed entirely of ions, presenting a wide range of solvent properties [18,19]. These characteristics offer possibilities for new ILs in chemical synthesis [20,21], catalysis [22], fuel cells [23], electrolytes [24], nanostructural organization [25] and new treatments for fibers [26–28], among other applications. Figure 2 summarizes the main applications of ILs. Despite their “green credentials”, ILs are versatile, although it is important to remember that not all ILs are environmentally benign [19,29].

Figure 2. Ionic liquids and their applications.
There are several types of IL, which can be classified according to the synthesis route and chemical structure. The most common ILs are 1-butyl-3-methylimidazolium chloride, 1-carboxyethyl-3-methylimidazolium bromide and 1-ethyl-3-methylimidazolium tetrafluoroborate. Hydrogen bonding is the main interaction of ILs with polysaccharides. Due to the presence of imidazolium C$_2$–H, C$_4$–H and C$_5$–H hydrogen atoms, the hydrogen bonding network between adjacent polysaccharide chains may be disrupted, especially the stronger acidic C$_2$–H. Then, cellulose derivatives may be modified, dissolved or partially dissolved by ILs [26,30].

The study of Polyelectrolyte is a growing field due to the vast range of applications in electrochemical (secondary batteries) and electrochromic devices (fuel cells, solar cells, supercapacitors and chemical sensors). Polyelectrolytes are defined as polymers in which the repeating units contribute an electrolyte group, forming a polycation and/or a polyanion. Usually, these groups dissociate into a solution, forming a conductive medium [31]. Some common examples of polyelectrolytes include DNA, glycosaminoglycans and polypeptides.

The main objective of the present study is to develop a systematic review to present, discuss and understand the advances in ionic liquids on polysaccharides published from 2014 to 2022. We focused on studies on the use of ILs as solvent media to obtain different compounds for use as electrolytes. A thorough description of the physicochemical properties and conduction mechanisms of ILs is beyond the scope of the work. We present a conclusion based on our findings, as well as residual challenges to be explored in the future. Our main findings include a summary of the most studied aspects in the field, as well as the easy-to-detect gaps in the existing literature.

2. Systematic Review Methodology

A systematic review is defined as research that uses the literature as a source of data, providing a summary of the evidence related to a specific intervention strategy by applying explicit, systematic and critical evaluation, as well as information synthesis, about a specific topic [32,33]. Briefly, a systematic review follows a methodical approach, also called a protocol, the “Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) [34]. Practice guidelines are established, and data of past and current studies are compiled, guiding future research efforts. When the protocol is followed, the correct key words and inclusion criteria are established. Thereby, omissions and biases are avoided, resulting in a more significant review with respect to the specific research topic [35]. In contrast to a conventional review, exclusion and inclusion criteria are employed with respect to the studies reviewed, and no arbitrary search is conducted when the protocol is followed. The methodology is presented below.

Three different literature databases were selected (Scopus (www.scopus.com, accessed on 18 January 2022), Web of Science (www.webofknowledge.com, accessed on 18 January 2022) and Scifinder (www.scifinder.cas.org, accessed on 18 January 2022)) to search for papers. For the search, the following terms were used: ([ionic liquid] AND [polysaccharide]). The search included papers published from 2014 to 2022.

Only papers related to the use of ILs as solvent media for polysaccharides were included in which fuel cells, batteries, sensors, metal plating or solar cells were considered and described.

3. Data Collection Results

Figure 3 shows the search and selection process diagram, following the PRISMA protocol, including the identification, screening, eligibility and included steps. A total of 472 papers were identified, including all research types, excluding conferences and review papers, among others. The next step was to exclude papers in which the word ionic liquid appeared but that were not related to polysaccharides or vice-versa, resulting in the exclusion of 103 research papers.
Figure 3. Search and selection process diagram, following PRISMA protocol.

Following exclusion of duplicate papers in the three databases, a total of 211 research papers were maintained. These final papers are related to the use of ILs and polysaccharides in different applications, such as synthesis, separation media, characterization, green solvents and electrolytes.

Due to the large number of studies in several different areas, we chose a specific topic (in our case, electrolytes) and focused on all papers published that met our search criterion. A total of 10 papers were identified and analyzed separately in the present work. Figure 4 presents all the searches conducted in the Scopus and Web of Science databases.

Figure 4. Results of searches of Scopus and Web of Science databases.

4. Results and Discussion

Table 1 summarizes all the papers selected using the systematic review protocol. We selected only papers regarding electrolytes.
Table 1. The main polysaccharides, reagents and methodology/application of selected studies.

| Reference | Polysaccharides | IL | Methodology/Application |
|-----------|-----------------|----|-------------------------|
| [14]      | - Alginic acid from brow algae  
- Pectin from citrus peel  
- Starch from potato | ● 1-butyl-3-methylimidazolium chloride | Methodology: preparation of hydrochars, ionochars and carbon materials in IL using an autoclave under autogenous pressure, as well as further washing and drying of the prepared material.  
Application: Li-ion batteries (LIBs) |
| [36]      | - Levan | ● Choline monocarboxylate IL | Methodology: dry casting of a solution mixed with levan and IL.  
Application: organic transistors. |
| [37]      | - Curdlan | ● 1-carboxyethyl-3-methylimidazolium bromide | Methodology: preparation of luminescent films using mixtures of Cur-NH$_2$, Cur-NH–IL or Cur–NH–SA dissolved in water with the appropriate amount of PVA.  
Application: luminescence sensors. |
| [38]      | - Catalpa  
- Indian Rosewood  
- Chinaberry  
- Babool | ● 1-butyl-3-methylimidazolium chloride | Methodology: pretreatment + hydrolysis  
Application: one-spot conversion of biomass to value-added products. |
| [17]      | - Potato starch | ● 1-methylimidazole acetonitrile | Methodology: synthesis and preparation of cationic starch gel polymer electrolytes.  
Application: dye-sensitized solar cells. |
| [39]      | - Chitosan  
- Iota-carrageenan  
- Curcumin | ● 1-methylimidazolium hydrogen sulfate | Methodology: precipitation of miscible solutions.  
Application: cytocompatibility. |
| [40]      | - Hexagonia apiaria fungus | ● 1-ethyl-3-methylimidazolium tetrafluoroborate | Methodology: mixing with KOH and further treatment of mixtures at 800 °C in a nitrogen atmosphere.  
Application: electrochemical capacitance. |
| [41]      | - Agar | ● 1-methyl-3-propylimidazolium iodide | Methodology: electrolytes prepared by dissolving of chemicals and gelatinization, as well as dye-sensitized solar cells prepared by coating with two layers of TiO$_2$.  
Application: dye-sensitized solar cells. |
| [42]      | - κ-Cg in powder form  
- (kappa- carrageenan) | ● 1-butyl-3-methylimidazolium chloride | Methodology: solvent casting of solutions containing IL, kappa-carrageenan, glycerol and water.  
Application: electrochemical devices. |

Figure 5 presents the different types of polysaccharides found in our search; the number on the graph represent the number of identified studies concerning a given polysaccharide. All cyclic chemical structural forms are presented. Some components present more than one monomer in nature, such as chitosan with acetylated and non-acetylated components and carrageenan with kappa, iota and lambda monomers. Furthermore, Table 1 summarizes and describes the reviewed papers in further detail, as well as the main reagents and process considered.

Baccour et al. (2020) [14] studied the decarbonization of polysaccharides into FeCl$_3$/1-butyl-3-methylimidazolium chloride (BmimCl) IL with the aim of synthesizing carbon with tunable textural and structural properties. The main results indicate that the FeCl$_3$/BmimCl molar ratio impacts nanoporosity and external pore volume. Chitosan yields carbon-negative electrodes with improved electrochemical properties, and external per volume nitrogen content boosts the electrochemical performance. According to the
authors, the best electrochemical performance was obtained with components prepared using IL/chitosan and “the lowest Fe/Cl₃/BmimCl ratio”. High pore volume ratio was also obtained, and the residual iron (Fe(0)) “prevail over the degree of graphitization in boosting the electrochemical properties of carbon materials, with potential applications in the field of energy storage and conversion.”

Figure 5. Result of searches of Scopus and Web of Science databases regarding polysaccharides.

Jo et al. (2020) [36] studied biocompatible and biodegradable organic transistors using a solid-state electrolyte incorporated with choline-based ionic liquid and polysaccharides. Figure 6 illustrates the main findings of this study. The unique properties of the electrolyte make it softer and more flexible, exhibiting electrochemical ion-transporting behavior related to the operation of organic transistors. The authors also fabricated organic transistors with a simple solution process and transfer printing method with new biocompatible and biodegradable ion-conducting solid-state electrolytes with water-soluble properties. The developed devices can be used for biomedical applications by fabricating more complicated logic circuits. The main finding revealed that the organic transistors can operate under low voltage conditions (−2.0V). Moreover, “high on/off ratio, negligible hysteresis, and mechanical reliability, bending by a variety of effect strains” were obtained. Electrocardiogram signals from humans were also measured using biodegradable organic transistors. The devices were attached directly to human skin, which enabled more efficient signal acquisition compared to conventional electronic devices.

Sun et al. (2020) [37] studied various flexible films as luminescent sensors for based acid vapor, as shown in Figure 7. The study was focused on the preparation and luminescence performance of the curdlan derivatives matrix and europium (III) complexes. According to the authors, the biopolymer matrix significantly enhanced the luminescence intensity and stability of the europium (III) nanohybrids, achieving a luminescent quantum efficiency of more than 50% with the flexible films. Another essential characteristic is that the produced films were colorless and transparent in sunlight and under a 365 nm ultraviolet lamp emitting a bright, red light. Moreover, when exposed to triethylamine vapors, the emission intensity increased sharply, and the opposite was observed when exposed to hydrogen chloride vapor. Hence, high sensitivity to both base and acid vapor was achieved.
Figure 6. ECG recording with the biodegradable organic transistors. Details of the organic transistor: (a) Schematic illustration of the biodegradable organic transistor with biodegradable LSE as substrate and dielectric, P3CPT as organic semiconductor and an Au electrode. (b) Equivalent circuit for measurement of ECG signals by the biodegradable organic transistors from the biological interface. (c) Photograph of the device on human skin (scale bar: 2 cm) and the heart (scale bar: 1 cm) attached without any other adhesives. (d) ECG signals recorded from human skin with standard equipment and the biodegradable organic transistors with VGS = 0 V (black) and VGS = −1.0 V (red) when VDS = −1.0 V with axes of time and current. (e) Change in ECG signal trace the day after fabrication. (f) Photograph of the device on a rat heart (scale bar: 1 cm). (g) ECG signals recorded from rat hearts with VGS = 0 V (black) and VGS = −1.0 V (blue) when VDS = −1.0 V with axes of time and current. (h) ECG signals from rat hearts recorded with the biodegradable organic transistors when VGS = −1.0 V and VDS = −1.0 V, showing the transience of trace from degradation by body fluid of the rat. “Reprinted from [36] Copyright (2020), with permission from John Wiley and Sons”.

Figure 7. (a) Luminescence effect photographs of the solution and films under a 365 nm UV lamp at different times. (b) Emission spectra obtained by 350 nm UV excitation of acid–base-sensitive films. (b1) EuTL@Cur-NH2, (b2) EuTL@Cur-NH-IL and (b3) EuTL@Cur-NH-SA. Inset: The corresponding photographs of films under 365 nm UV light (from left to right the films are expose to Et3N, expose to air and expose to HCl). “Reprinted from [37] Copyright (2020), with permission from Elsevier”.
Tyagi et al. (2019) [38] studied the simultaneous pretreatment and hydrolysis of hardwood biomass species catalyzed by a combination of modified activated carbon and ionic liquid in a biphasic system. The developed methodology facilitated the one-pot conversion of hardwood biomass to value-added products. The developed process enhanced the conversion due to the addition of electrolytes to the system without a requirement for separate delignification. According to the results, “one-pot conversion of hardwood biomass was most effective using a combination of acid and ionic liquid for pretreatment and subsequent hydrolysis”. Aluminum chloride enabled afforded the strongest Lewis acid at 4 wt.%, which increased the yield to 96.56% (total reducing sugars) and 86.23% 5-hydroxymethylfurfural. Moreover, the addition of dimethyl sulfoxide (DMSO) improved the partition coefficient at an optimized ratio. Finally, the reaction system was recycled six times with a minimal loss of catalyst.

Lobregas and Camacho (2019) [17] applied a dye-sensitized solar cell with a synthesized quasi-solid-state gel polymer based on potato starch (Figure 8). The polysaccharide was also grafted with imidazolium-based ionic liquid. Different cationic starch: ionic liquid (CS:IL) ratios and wt.% of KI/I2 redox couples were tested until optimization. The optimized gel polymer electrolyte presented a CS:IL ratio of (1:3) and a 70% pf wt.% of KI/I2. These proportions guarantee a gel with the highest conductivity and most efficient ion migration. Briefly, the electron migrates into the gel matrix to form an interconnected network between the tiny spaces of adjacent TiO$_2$ nanoparticles, which aids in the migration of the electrons. According to the authors, the main drawback was poor performance in terms of liquid electrolyte control, although relative stability was obtained. Last but not least, a dye-sensitized solar cell was fabricated, achieving 0.514% efficiency.

Ramin et al. (2019) [39] studied iota-carrageenan curcumin-based materials by precipitating miscible solutions prepared in IL. A polyelectrolyte complex (PEC) loaded with curcumin (CUR) was prepared by precipitating polymeric/CUR solutions prepared in methylimidazolium hydrogen sulfate [Hmim][HSO$_4$]. According to the authors, “The ionic liquid solvent use enables an in situ encapsulation method, promoting high encapsulation efficiency of CUR in the PEC (ca. 95%).” The authors related that “the residual ionic liquid in the PECs must be removed because it contributed cytotoxicity to mammalian “Z cells (adipose-derived mesenchymal stem cell (ADSCs) and bone marrow-derived mesenchymal stem cells (BMSCs))”.

Deng et al. (2017) [40] showed the enhancement of electrochemical capacitance of biomass carbon by pyrolysis of extracted nanofibers. Polysaccharide nanofibers were extracted from hexagonal apiarian (HA) fungus through a hydrothermal method using 1-ethyl-3-methylimidazolium tetrafluoroborate (EMIImBF$_4$). According to the authors, “porous carbons, derived from the extracted nanofibers, exhibited a two-fold increase in electrochemical capacitance than those derived directly from the crude HA, tested in potassium hydroxide aqueous solution”. This was due to the larger surface area and a higher structural order. Furthermore, the capacitances for both porous carbons in the IL electrolytes were lower than in aqueous electrolytes, and the decay of capacitance at a higher discharge current was even more severe.

Nadia et al. (2016) [41] studied quasi-solid-state agar-based polymer electrolytes (QSPE) for dye-sensitized solar cell applications using imidazolium-based ionic liquid. Two iodide salts (sodium iodide and potassium iodide) were used for the first two systems. QSPE with 50 wt.% potassium iodide was incorporated with 1-methyl-3-propylimidazolium iodide (MeC$_4$ImI) IL for the third system. The highest ionic conductivity of 1.48 $\times$ 10$^{-3}$ S cm$^{-1}$ was achieved with the addition of 3.0 g of MeC$_4$ImI. Dye-sensitized solar cells were fabricated by sandwiching QSPEs between the counter and working electrodes. The AKP-5 sample with 3.0 g of MeC$_4$ImI showed the highest energy conversion efficiency of 2.16%.
Nunes et al. (2017) [42] studied electrolytes using red seaweed for electrochemical devices (Figure 9). The obtaining process was straightforward and environmentally friendly: green electrolytes composed of kappa-carrageenan, 1-butyl-3-methylimidazolium chloride ([Bmim]Cl) IL and glycerol were incorporated in an aqueous solution. The authors produced membranes without lead-to-water formation and without requiring the flow of gases. The membranes were predominantly protonic conductors, with potential applications including flexible, high-performance energy storage devices; supercapacitors; batteries; and electrochromic devices. According to the authors, high levels of ionic conductivity were reached due to the complete dissociation of [Bmim]Cl in the electrolyte medium.
In this study, we reported a systematic review of literature published between 2014 and 2022 of the most recent advances in understanding, as well as residual challenges, associated with ionic liquids on polysaccharides. Electrolytes have not been extensively explored, given that only 10 papers identified on this topic. In the present study, we summarized the numerous methods and applications available in the literature, demonstrating the versatility of ionic liquid applications, including as biodegradable electrolytes and organic transistors. Many methodologies have been reported in the literature for the manufacture of such materials. Solvent casting, dry casting and autoclaving are among the reported techniques; however, many methods have been reported, depending the final application. Li-ion batteries (LIBs), solar cells, high-performance energy storage devices as supercapacitors and batteries and electrochromic devices, among others, are the main applications of these newly developed materials. In this systematic review, we reported on the application of polysaccharides combined with ionic liquids and other reagents, as well as their applications, with the aim of compiling data for use in future applications.

6. Future Prospects

Societal pressure regarding environmental appeal is increasing, leading researchers to develop materials based on green chemistry as efficient as less eco-friendly materials. The development of polysaccharide-based electrolytes opens the possibility of adapting to the aforementioned appeal by society.

Different types of ILs combined with polysaccharides to produce electrolytes showed different levels of efficiency, improving the final electrolyte properties. However, selection of IL and polysaccharide types is subjective. In this sense, this systematic review pointed out the gaps in the literature.

Among the different polysaccharides listed in this study, the most used as a base for electrolytes are carrageenan, curcumin and starch, with distinct ILs. Hence, future research
should focus on different polysaccharides, such as microcrystalline cellulose, chitosan and biomass components (cellulose, hemicellulose and lignin).

Electrolytes have a continuous market for high-voltage and safe applications in advanced lithium batteries. Chen et al. (2020) reported on recent progress concerning liquid-to-solid batteries. The main challenges include safety, rate capacity and energy density, for the vehicle industry. The authors reported that, in the future, the inorganic solid electrolytes may be applied in power batteries for vehicles and large-scale power grids.” They also commented that polymer electrolytes may be assembled in flexible batteries for flexible screens, wearable devices and other 3C products.

Finally, the use of polysaccharide-based electrolytes in combination with ILs is not yet practical (in general) in industrial terms, and improved knowledge of their processing is required.

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