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Review

Biopolymer-Based Hydrogels for Harvesting Water from Humid Air: A Review

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Abstract: Despite the shrinkage of water availability worldwide, the water demand has been climbing due to the rapidly increasing human population and the impacts of numerous hydroclimatic factors. To feed the growing population and meet with the exigent need for freshwater, new water sources must be identified and utilized. In recent years, biopolymer-based hydrogels have been explored as a sustainable and inexpensive solution to capture moisture from humid air for water supply. This review summarizes recent advances in the preparation of atmospheric water harvesting hydrogels that are synthesized based on cellulose, chitosan, and/or other biopolymers, as well as their agricultural applications. The properties and performance of different biopolymer-based hydrogels are discussed and compared. Since agriculture accounts for more than 70% of the water consumption, developing a cost-effective hydrogel-based atmospheric water harvesting/supply system could be a promising solution for relieving the elevating water stress in arid and semi-arid regions.

Keywords: biopolymer; cellulose; chitosan; hydrogel; starch; moisture capture; relative humidity; water harvesting

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1. Introduction

Fresh water is a finite resource in escalating demand [1]. With the rapidly increasing human population worldwide and continually expanding industrialization in many countries, more fresh water is needed for the prosperity and survival of mankind in the following decades [2–4]. While water demand is increasing, water availability is shrinking [1]. The excessive withdrawal, aquatic pollution, changing climate, and many other complex factors have added more challenges for keeping water demand below its availability [4,5]. According to the projections by many sophisticated models, over 6 billion people may suffer from water shortages by 2050 [6–8]. To combat the decreasing water availability, new water sources must be exploited. Moreover, water use efficiency needs to be improved to reduce waste.

Water exists in the atmosphere in its gaseous form. According to the law of thermodynamics, with a 1 °C increase in the air temperature, the relative humidity (RH) in the air shall increase by approximately 7% [9]. As per the observations over the past several decades, the air in most parts of the world has become increasingly humid [10]. Given the significantly increased and continuously increasing moisture in the air, decentralized atmospheric water harvesting could be a plausible and sustainable solution for relieving the heightening water stress in many places, especially in rural, remote, and landlocked areas. Unlike desalination technologies that require access to brackish water, high energy supply, and expert labor for operation and maintenance [11], atmospheric water harvesting is easily accessible everywhere and can be integrated with a renewable energy source (e.g., solar energy) for local needs [12]. Because the water sourced from atmosphere is mostly clean, oftentimes there is no need of further treatment for the harvested water to be used in agriculture or industry [12].

Current technologies for atmospheric water harvesting can be categorized into (1) direct harvesting via condensation, e.g., fog and dew water collection, (2) vapor concentration via membrane or desiccant, e.g., membrane separation and sorbent sorption, and (3) byproduct collection via integrated systems. Unfortunately, few of the existing technologies have been commercialized, mainly due to the challenges related to their cost-effectiveness, scalability, and/or stability [13]. For example, in passive radiative condensers, although the theoretical maximum dew yield through dew water collection is 0.8 kg/m^2/day according to the radiative cooling ability available for condensation, the actual maximum yields recorded in arid and semi-arid areas were in the range of 0.3–0.6 kg/m^2/day [14]. The above difference is greatly affected by factors such as the radiative heat exchange rate, weather conditions, and material properties [14]. Moreover, the water use efficiency and energy efficiency will further decrease when the collected atmospheric water is distributed and supplied for agricultural or industrial purposes [15]. For the commercialization of atmospheric water harvesting, novel approaches towards the direct utilization of water captured from humid air are urgently needed.

A recent U.S. patented method (No. US 11310973 B2) provided a practical solution for lowering agricultural water demand by sparging humid air to the root zone and capturing/storing atmospheric water with superabsorbent hydrogel, a crosslinked hydrophilic polymer [16]. Many studies have shown that, when saturated, superabsorbent hydrogels are able to absorb and hold water up to 1000 times their dry weight [17,18]. In an agricultural field, when the moisture of the root zone soil decreases, the captured water can be slowly released as driven by the matric potential difference, thus wetting the soil and providing water for plants [19]. As shown in Figure 1, a subsurface dripline can be adapted to deliver humid air towards the hydrogels mixed with the root zone soil. A solar powered air pump can be integrated to minimize the energy requirement for the system.

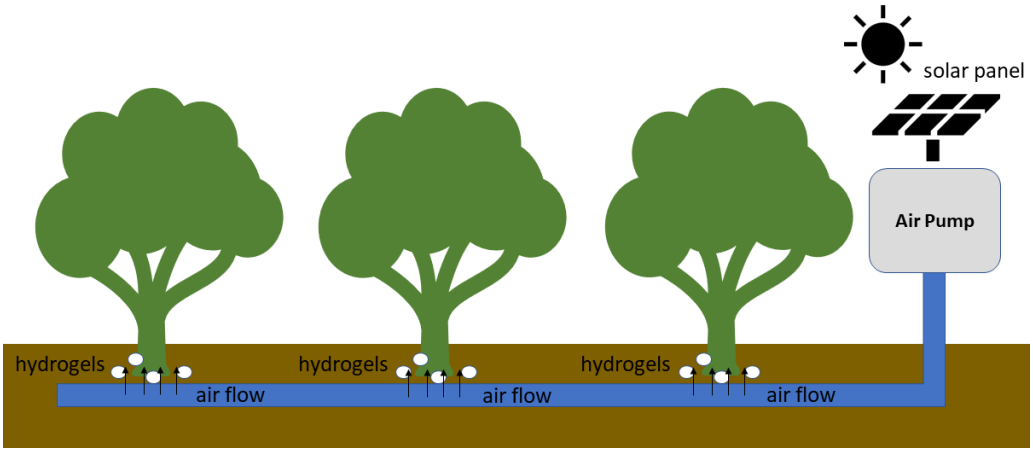

Figure 1. Hydrogel-based solar-powered atmospheric water harvesting and supply system.

The most widely used hydrogels as a soil amendment used in agriculture are based on polyacrylamide and/or polyacrylic acid. Cheng et al. synthesized a poly(acrylic acid-co-acrylamide) composite hydrogel with a maximum water absorption ratio of 494 g/g [20]. When applied to agricultural fields, these hydrogels were able to ameliorate soil permeability, improve water penetration rate, and enhance water retention in arid and semiarid areas where the supply of water for irrigation is under stress [21,22]. However, the applications of both polyacrylamide- and polyacrylic acid-based have been controversial because of the ecological and public health concerns on their moderately toxic intermediate products generated during their natural degradations [23,24]. To address these issues, biodegradable and environmentally friendly hydrogels that are based on biocompatible compounds (e.g., cellulose, chitosan, starch, and alginate) and their derivatives have been explored for the synthesis of hydrogels for agricultural applications [25].

Although there have been many reviews focusing on the preparation, characterization, and applications of biopolymer-based hydrogels, few of them have given prominence to hydrogel's potential for atmospheric water harvesting. The efficacy of the atmospheric water irrigation system shown in Figure 1 heavily relies on the applied hydrogel's effectiveness in capturing water from the humid air pumped into the system. Therefore, it is critical to summarize and compare the synthesis process, swelling behavior, water absorption capacity, and other physicochemical properties of various types of superabsorbent hydrogels developed over the recent years. While many existing reviews have completed such work regarding the prevalent non-biopolymer-based hydrogels for various applications, the focus of this current review is on biopolymer-based hydrogels for atmospheric water harvesting.

2. Non-Biopolymer-Based Hydrogels

Although non-biopolymer-based hydrogels have been criticized for their inability to completely biodegrade in the environment and their toxic intermediate products during their slow degradations, these hydrogels often have superior interactions with water molecules, which allow them to capture water vapor efficiently, convert vapor to liquid rapidly, store water in large quantities, and release water to the surrounding under different weather conditions. For example, in a recent study, hygroscopic polypyrrole chloride was integrated into the hydrophilicity-switchable polymeric network of poly N-isopropylacrylamide [26]. At a low RH of 30%, the hydrogel was able to absorb and store water at approximately 23 g/g. The captured water could be released by half within the first 20 min when the temperature was raised to 40 °C, and then gradually release at about 0.3 g/g/h through an evaporating–condensing process [26]. In a subsequent field study, the effective water transport along the interpenetrating networks of functional polymers enabled the flexible adaptation of this hydrogel to the robust soil environment in agriculture [26]. In another study, for the enhancement of water vapor sorption capacity and swelling properties, poly [2-(methacryloyloxy)ethyl]dimethyl-(3-sulfopropyl)ammonium hydroxide (PDMAPS) hydrogels were loaded with hygroscopic LiCl salt. At a low RH of 30% in a custom-made system, the PDMAPS-LiCl hydrogels reached equilibrium after about 120 min and were able to produce clean water at a daily rate of 5.87 L/kg [27]. Similarly, different clays (e.g., laponite, bentonite, and palygorskite) have been explored as additives to hydrogels of sodium acrylate and acrylamide [28]. The synergy due to the porous polymeric structure of hydrogels and the hydrophilicity of clays significantly improved the performance and reusability of the integrated polymer desiccants at different levels of relative humidity [28]. In general, when hygroscopic salts (e.g., LiCl) were incorporated into the matrixes of hydrogels, the atmospheric water harvesting performance of the composites could be significantly enhanced. However, the dissolution of these salts in water is a problem that needs to be addressed. Some recent studies integrated biochar or carbon nanotubes (CNTs) to the sorbents, which stabilized the hygroscopic salts and enhanced the swelling capacity [29–31]. In one of these studies, the CNT-hydrogel composites synthesized via vacuum drying were capable of harvesting atmospheric water when the relative humidity was below 20% [31]. Photothermal material (i.e., graphene) and deliquescent salt (i.e., $CaCl_2$) have also been explored to integrate with polyacrylonitrile (PAN) and acrylamide (AM) for the formation of a hybrid hydrogel, which was able to generate 1.04 L of freshwater per kg of hydrogel at a low RH of 60% [32]. Although non-biopolymer-based hydrogels have various advantages in terms of their water swelling performance, their unsustainable nature does not align with the current trends in material science and engineering towards being renewable and green. As a result, the use of bio-based materials for hydrogel synthesis has attracted much recent attention.

3. Hydrogels Based on Cellulose and Its Derivatives

Cellulose is one of the most abundant organic compounds on the Earth. It is renewable, biocompatible, and biodegradable. Owing to the high inter- and intramolecular hydrogen

bonds and van der Waals forces, the hydrophilicity and resistance to dissolution make cellulose and its derivatives ideal materials for making atmospheric water harvesting hydrogels [33]. Hydrogels that are prepared based on cellulose and its derivatives typically exhibit good mechanical properties and minimal environmental impacts.

Dissolution is required in order to expand the applications of cellulose for hydrogel synthesis. Commonly used solvent systems for cellulose dissolution include but are not limited to NaOH/urea, NaOH/thiourea, LiOH/urea, and ionic liquids. The level of homogeneity of cellulose in the solution depends on the type of solvent system selected, solvent temperature, stirring rate, stirring time, and the molecular weight and crystallinity of cellulose [33]. In general, the combination of alkali and urea (or thiourea) results in better dissolution than alkali alone. Through different chemical reactions, various cellulose derivatives can be obtained and then be used for hydrogel synthesis, which is another popular approach to widen the applications of cellulosic materials [33].

Hygroscopic salts (e.g., LiCl, $CaCl_2$, and $MgCl_2$) have been integrated into cellulose-based hydrogels to improve their ability to capture moisture. For example, as shown in Table 1, a recent paper reported adding LiCl and 1% graphene (by weight) to a hydrogel synthesized with wood-derived nanofibrillated cellulose as its hydrophilic skeleton [34]. This hydrogel was able to rapidly absorb a large quantity of atmospheric water, even in severely dry climate conditions (e.g., low RH of ~18%) with very little energy from natural sunlight (0.10–0.56 kW/m^2) for solar-driven vapor evaporation [34]. In a similar study, cellulose and hemicellulose were extracted from waste corn stalk for the fabrication of a LiCl-coated hydrogel. Owing to the significantly porous structure and highly hydrophilic nature of the hydrogel, effective atmospheric water harvesting up to 1.8 kg/kg was observed when the RH was shifted between 20 and 80% [35]. In addition, through the surface modification of the material using commercial carbon ink, due to the moisture gradient and negative surface charge, the directional hydronium migration along the micro-channels within the material was stimulated, leading to the induction of an electrical voltage up to 0.6 V while harvesting atmospheric water [35]. In another recent study, LiCl was mixed with konjac glucomannan (KGM) and hydroxypropyl cellulose (HPC) to prepare the biopolymer-based hydrogel via solution polymerization and liquid nitrogen cooling [36]. Throughout 14 to 24 cycles per day in arid environments, the hydrogel was able to yield fresh water at 5.8 L/kg and 13.3 L/kg when the RH was 15% and 30%, respectively [36]. Although hygroscopic salts significantly enhanced the hygroscopicity of hydrogels, passivation layers could be formed due to the agglomeration of salt crystals during hydration. As a result, the sorption kinetics may quickly slow down over time, resulting in the deterioration of the hydrogel's cycling performance [37]. By filling the hollow and interconnected fibers extracted from a loofah sponge with LiCl and replacing the original luffa peel with a bacterial cellulose (BC)/carbon nanotube (CNT) photothermal conversion membrane, Yao et al. created a hydrogel with an excellent water absorption capacity of 2.65 g/g at 90% RH and fast water release at 1.33 kg/m^2/h under 1.0 sun [38]. As shown in Figure 2, the LiCl-filled hollow fibers were able to attract and store moisture from the ambient air, and the interconnected internal channel could transport water for its release to combat water stress.

Porous natural absorbents (e.g., bentonite and zeolite) have also been mixed with cellulose-based hydrogels to enhance water absorption and retention. A recent study used the Michael Addition reaction to modify cellulose. Carbamoyl ethyl ether of cellulose (CEEC, as a precursor) was obtained before mixing with bentonite in a solution to produce the hydrogel [39]. Rapid water swelling was observed as the gel-soil mixture (5% clay) had 19,040% swelling within 26 min at 25 °C and 36,600% at equilibrium after approximately one week [39]. During the pot experiments conducted in semi-arid conditions (32 °C, 90% RH), the groups of *Brassica juncea* in hydrogel-containing black soils were observed to be more resilient than the controls, in terms of water use, seedling, and survival rate [39]. Other similar studies also reported significant improvement in water retention, plant growth and biomass yield [28,40,41].

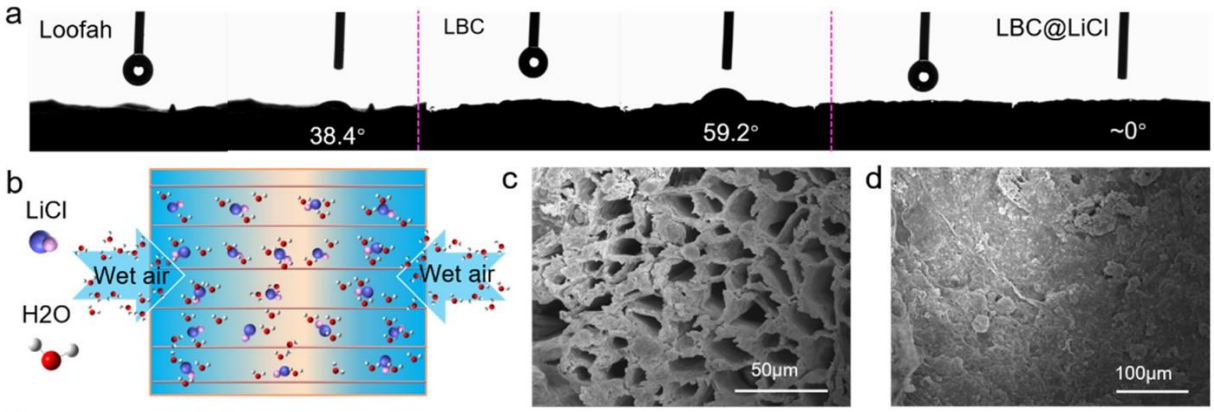

Figure 2. (**a**) Water contact angles of loofah sponge, loofah/BC/CNT hydrogel, and loofah/BC/CNT hydrogel filled with LiCl; (**b**) schematic of loofah fiber channels storing LiCl and absorbing moisture from air; (**c**) scanning electron microscopy (SEM) of the loofah fiber cross section; and (**d**) SEM of BC/CNT epidermis treated with a 20% LiCl ethanol solution. Reprinted with permission from Yao et al., 2022 [38].

Table 1. Summary and comparison of various hydrogels based on cellulose and its derivatives.

Biopolymer Type	Additive(s)	Relative Humidity in the Environment	Water Harvested/Generated per Unit Weight of Hydrogel (g/g)	Reference
Nanofibrillated cellulose	Graphene (1%) and LiCl	18–95%	0.3–2.36	[34]
Cellulose and hemicellulose	LiCl	20–80%	0.46–1.84 *	[35]
Hydroxypropyl cellulse and konjac glucomannan	LiCl	15–30%	5.8–13.3 *	[36]
Bacterial cellulose	Carbon nanotube	90%	2.65	[38]
Cellulose acetate	Poly(N-isopropylacrylamide)	7–90%	Up to 20.8 *	[42]

* Unit has been converted into g/g from the originally reported unit for the purpose of comparison.

Novel polymerization methods have also been developed in recent years for the synthesis of cellulose-based hydrogels to be more functional across a broad range of relative humidity. Through a novel shell electrospinning method, Thakur et al. fabricated a temperature-responsive hydrogels based on cellulose acetate (CA) and poly(N-isopropylacrylamide) (PNIPAM), which could rapidly absorb atmospheric water (i.e., being hydrophilic) at ambient temperatures and effectively release water (i.e., being hydrophobic) at higher temperatures [42]. The CA-PNIPAM hydrogels demonstrated a maximum moisture uptake capacity of 208 ± 6% across a broad range of temperatures (25–45 °C) and RH (7–90%) [42]. The temperature-responsive property of PNIPAM enabled the efficient capture and release of moisture and made this novel hydrogel promising for arid regions, where the external temperature stimuli can be provided by the continuous natural day and night temperature cycles.

4. Hydrogels Based on Chitosan

Chitosan is a linear polysaccharide consisting of randomly distributed β-(1→4)-linked D-glucosamine (i.e., the deacetylated unit) and N-acetyl-D-glucosamine (i.e., the acetylated unit). It can be made by treating the chitin shells of shrimp and other crustaceans with an alkaline substance (e.g., NaOH). In fact, chitosan is one of the most plentiful biopolymers, only secondary to cellulose. Besides being abundant, inexpensive, biocompatible, and biodegradable, chitosan-based hydrogels have demonstrated considerable enhancement in the structural durability upon repeated use [43]. One of the major challenges with most of the chitosan-based superabsorbent hydrogels is the release of condensed water back to the environment. Among the different methods for desorbing water (e.g., heating, pressurization, solar radiation), solar-driven evaporation is gaining increasing popularity because of its sustainable features.

Chitosan-based hydrogels have demonstrated great potential for their applications as hydrogel evaporators. As shown in Figure 3, a molybdenum carbide/carbon-based chitosan hydrogel (MoCC-CH) was recently designed for effectively absorbing atmospheric water and also efficiently accelerating water evaporation by tuning the proportion of the chitosan matrix in the hydrogel [44]. The MoCC-CH was able to achieve a high solar-thermal conversion efficiency of 96.15% under one sun illumination and yielded 13.86 kg/m^2 of fresh water during the outdoor experiments under sunny conditions [44]. Fan et al. synthesized a high-performance hydrogel by mixing chitosan into gelatin-polyvinyl alcohol hydrogels through gamma ray induction. The tensile strength, swelling properties and water evaporation rate of this composite has been significantly improved [45]. In another recent study, the integration of poly(N-acryloyl glycinamide) PNAGA, chitosan, and carbon nanotubes (CNTs) resulted in a durable and efficient solar-driven evaporator, which had a solar absorbance as high as 97.2% and steam generation rate up to 2.42 kg/m^2/h [46]. Due to the large presence of –NH_2 functional groups in chitosan chains, the chitosan-based hydrogel evaporators showed strong resistance to bacterial growth and excellent tolerance to different robust environmental conditions (e.g., salty, acidic, or alkaline) [46].

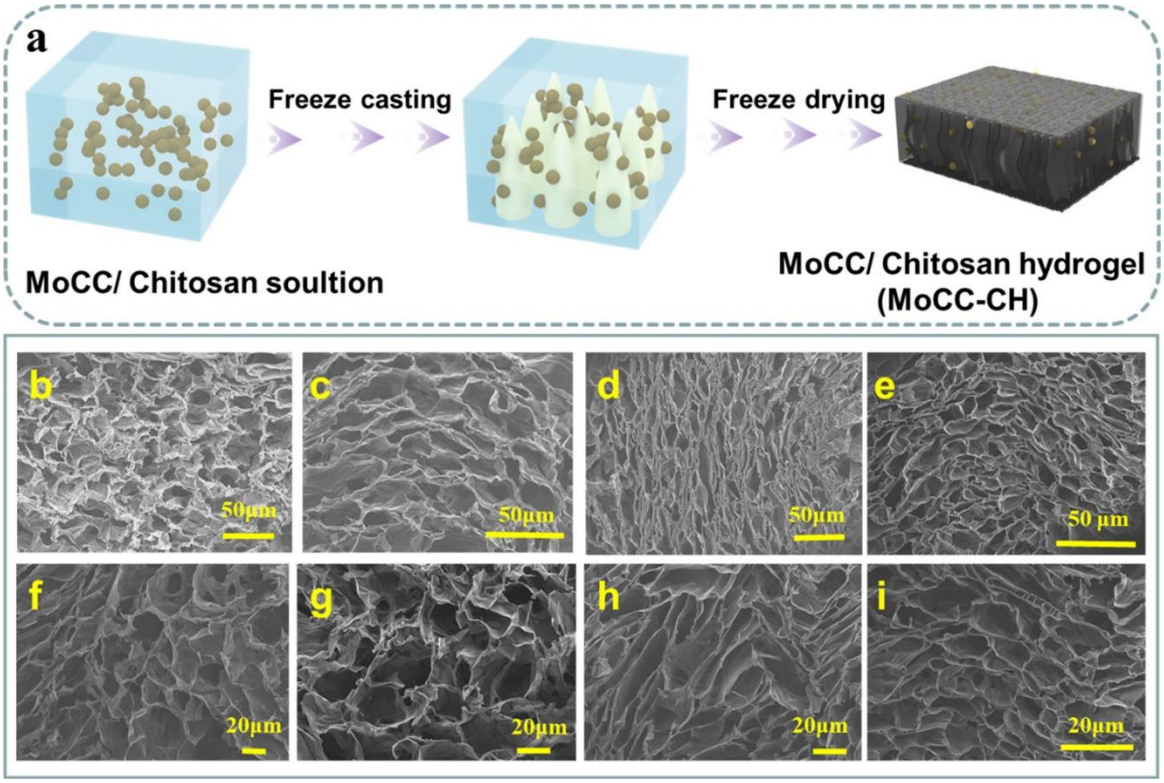

Figure 3. (**a**) Schematic illustration of the fabrication process of MoCC-CH via a freeze casting–freeze drying method; (**b**–**e**) SEM images of CH with different chitosan contents; and (**f**–**i**) SEM images of MoCC-CH with different chitosan contents. Reprinted with permission from Yu et al., 2022 [44].

Another popular area of application of chitosan-based hydrogels is fog harvesting. Through electrostatic spinning, Liu et al. developed chitosan-based heterostructure rough spindle-knot microfibers that could easily be modified to adjust their surface morphology, which subsequently affect the efficiency of fog harvesting [47]. The spindle-knot microfiber mainly consists of chitosan and sodium alginate. The former contains non-toxic cation polyelectrolyte with bacteriostasis, which may lower the risk of bacterial contamination in the collected water; the latter provides good fluidity and helps with the formation of ionic crosslinked gel with $CaCl_2$-Ca^{2+}, which creates electrostatic attractions with polyanionic

chitosan molecules [48]. The chitosan/$CaCl_2$ microfibers are scalable and have a great potential for large-scale and directional water collection [47].

5. Hydrogels Based on Other Biopolymers

Besides the two most abundant biopolymers (i.e., cellulose and chitosan), many other biopolymers, such as starch, glycogen, chitin, and alginate, have also been explored for the synthesis of hydrogels for atmospheric water harvesting [49–51]. For the optimal water swelling capacity and structural stability, crosslinking is often needed through either physical or chemical processes. Physically crosslinked hydrogels contain different polymer chains connected through noncovalent physical interactions, including hydrogel bonding, coil–coil interactions, stereo complex formations, and ionic interactions [52]. For example, mannuronic acid and glucuronic acid in the structure of alginate can derive crosslinks with Ca^{2+}, thus crosslinking the polymers via the ionic interactions [53]. In chemically crosslinked hydrogels, in the presence of crosslinkers such as glutaraldehyde, N,N′-methylene-bis-acrylamide (MBA), divinyl sulphone, 1,6-hexanedibromide, or epichlorohydrin, various polymer chains are joined by irreversible chemical bonds to form the polymeric network [52]. Non-crosslinked hydrogels are more vulnerable to disintegration during the process of absorbing excess water (Figure 4). The crosslinked networks allow hydrogels to be stretched to a greater extent before failure. Although crosslinkers such as MBA may have limited biodegradability in soils, these chemicals usually pose little or no toxicological hazards due to their small dosage (e.g., <0.5% wt.) and low toxicity [54].

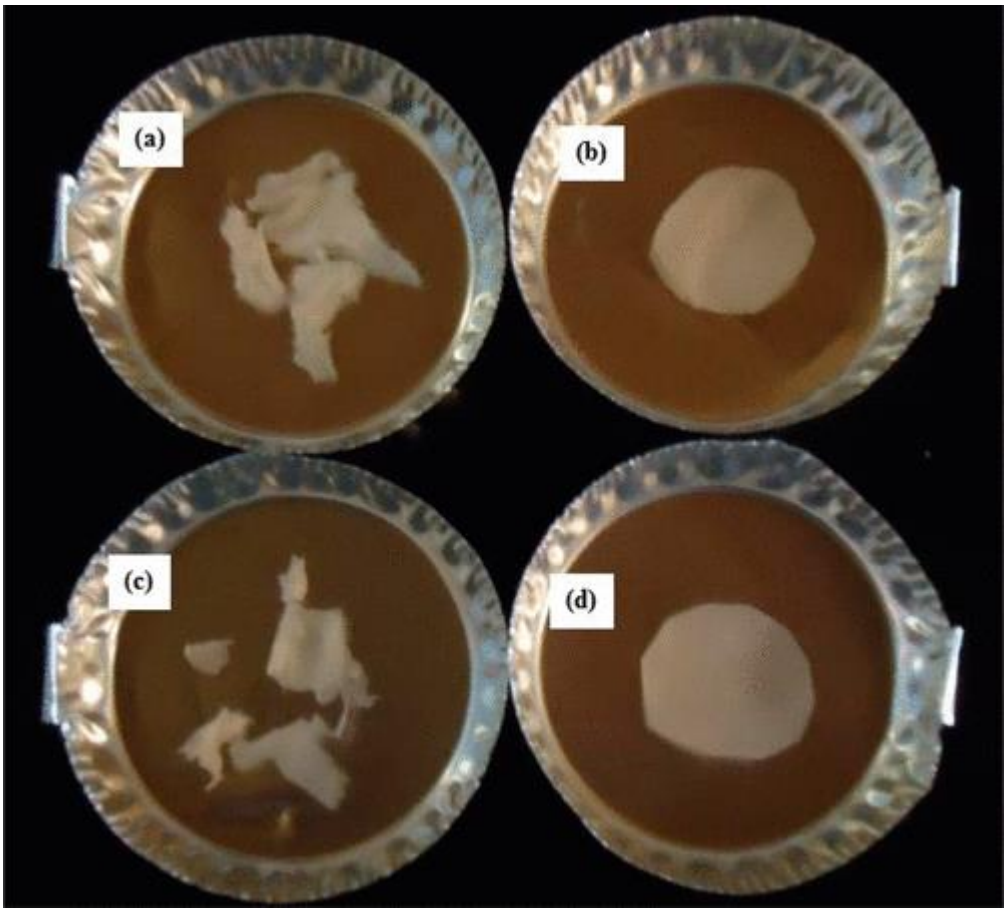

Figure 4. Non-crosslinked and crosslinked hydrogels after water immersion: (**a**) non-crosslinked hydrogel consisting of 25% softwood-derived nanofibrillated cellulose, (**b**) crosslinked hydrogel consisting of 25% softwood-derived nanofibrillated cellulose, (**c**) non-crosslinked hydrogel consisting of 25% hardwood-derived nanofibrillated cellulose, (**d**) crosslinked hydrogel consisting of 25% hardwood-derived nanofibrillated cellulose. Reprinted with permission from Nair et al., 2014 [55].

Many biopolymers have been integrated with synthetic ingredients for the formation of high-performance hydrogels for capturing moisture. For instance, a *Gum ghatti* and acrylamide mixture was initiated by $K_2S_2O_8$—ascorbic acid solution and crosslinked by MBA to prepare a 3D network hydrogel for water absorption. The product was able to selectively remove saline from different petroleum fraction–saline emulsions [56]. Using kelp as one of the raw ingredients, another research group developed a hydrogel with self-contained properties such as being hygroscopic, photothermal, temperature-responsive and durable [57]. At RH of 90%, the kelp-derived hydrogel was able to absorb more than 5.0 g/g of atmospheric water. Moreover, the absorbed water was able to be quickly released to sustain the plant germination and growth under a broad range of solar intensity (0.6–1.0 sun) and temperature (40–60 °C) [57].

6. Opportunities, Challenges, and Future Directions

With the increasing emphasis on the renewability, sustainability, and biocompatibility of materials used in agriculture, there has been an ascending trend in the number of studies on biopolymer-based superabsorbent hydrogels in the past decade, especially over the last couple of years (Figure 5). According to the literature search results on Wiley Online Library, the number of published articles about hydrogels that are mainly based on cellulose, chitosan, and/or starch with the purpose for atmospheric water harvesting have been continuously increasing over the past decade. The numbers of studies on cellulose- and chitosan-based hydrogels increased more than fourfold in less than ten years and nearly doubled in the last three years. The exponential increases in the numbers of studies on biopolymer-based hydrogels occurred in the last few years reflected the increased interest by governments to explore biopolymers as a substitute for synthetic polymers. As the circular economy gains more momentum as a new economic paradigm [58], there is likely to be more research support from both the government and the industry for the development of high-performance biopolymer-based superabsorbent hydrogels considering their versatile applications in various dimensions of the society in the near future.

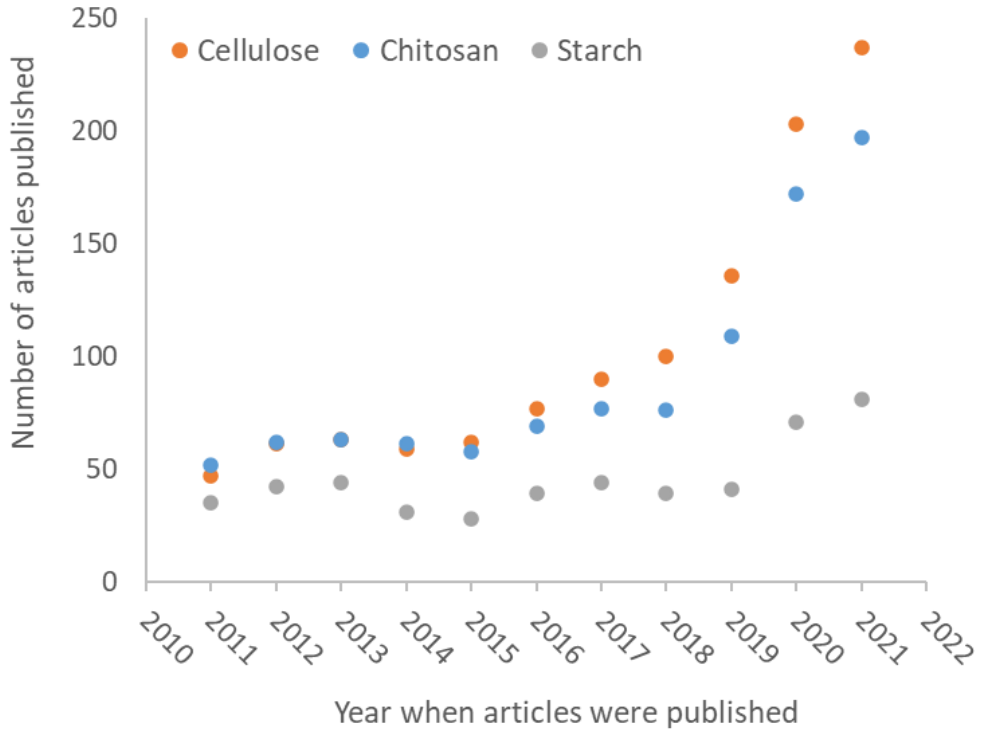

Figure 5. Number of articles about hydrogels based on cellulose, chitosan, and starch for atmospheric water harvesting from 2011 to 2021. The search was performed on Wiley Online Library, accessed on 5 November 2022.

At the same time, it should be noted that the total number of studies on biopolymer-based hydrogels purposed for atmospheric water harvesting is still small. Only 515 relevant articles were found in the Wiley Online Library for the complete year of 2021, indicating an underestimated and highly potential research area. While many studies show that biopolymer-based hydrogels produced at a low cost via simple methods can achieve high efficacy for various applications [59–61], conventional hydrogels based on polyacrylamide and/or polyacrylic acid may not decrease their market shares in a short time, especially as they have been prevalently applied in practice over the past decades. Although biopolymer-based hydrogels also have various advantages such as their abundant availability, renewability, easy synthesis, structural stability, and enhanced mechanical properties, it will take public education, economic incentives, and time to change the current consumer behaviors towards a more environmentally friendly direction [62].

In recent atmospheric water harvesting applications, many biopolymer-based hydrogels were able to present strong affinity to water vapors when the RH in the environment was high (e.g., >80%). However, the atmospheric water harvesting performance typically deteriorated quickly when the relative humidity level dropped below 30%. In addition, due to the reduced hygroscopicity of the hydrogels in arid environments, part of the harvested water vapors has been observed to evaporate fast. Future designs of biopolymer-based hydrogels for use in dry environments need to consider 1) improving the hygroscopicity at lower RH by exploring the integration with different hygroscopic additives and 2) decreasing evaporation rate to mitigate water loss via the assistance of biodegradable low-permeability coatings.

7. Conclusions

Being abundant, renewable, and biocompatible, biopolymer-based hydrogels for atmospheric water harvesting seem to be a promising sustainable solution to the global issue of water scarcity. Besides the two most plentiful biopolymers, i.e., cellulose and chitosan, many other biopolymers from diverse sources have been explored for the preparation of hydrogels with enhanced properties and lower costs. Technical breakthroughs are expected to improve the hygroscopicity and limit evaporation of water from biopolymer-based hydrogels under low-humidity high-temperature conditions.

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