Review

Superabsorbent Hydrogels in the Agriculture and Reclamation of Degraded Areas

Katarzyna Sroka [1],* and Paweł Sroka [2]

1 Faculty of Geo-Data Science, Geodesy, and Environmental Engineering, Department of Environmental Management and Protection, AGH University of Krakow, 30-059 Krakow, Poland
2 Faculty of Food Technology, Department of Fermentation Technology and Microbiology, University of Agriculture in Krakow, 31-120 Krakow, Poland; pawel.sroka@urk.edu.pl
* Correspondence: sroka@agh.edu.pl

Abstract: Superabsorbent materials (SAPs) are crosslinked polymer networks composed of ionic and non-ionic monomers. SAPs can absorb and retain water solutions up to several hundred times their own weight. As a result of swelling, they form a gel that is insoluble in water, considered safe and decomposes over time. This review focuses on the synthesis, degradation and chemical composition of SAP materials, with particular emphasis on chemical substances that are soluble in water and can migrate into the environment. Numerous applications of natural and synthetic hydrogels in agriculture and the reclamation of degraded areas in preventing erosion, retention water, reducing leaching of colloidal soil components and plant protection products, fertilisers and mineral salts into surface waters have been described. The influence of SAPs on the microbial activity of soils is described. New trends in the search for environmentally friendly SAPs made of modified biopolymers and waste materials are presented, which not only increase yields, but also ensure sustainable agro-environmental development.

Keywords: superabsorbent; hydrogel; swelling; polyacrylates; water retention; soil amendment; nutrient release; agriculture

1. Introduction

The escalation of extreme meteorological events, significant water shortages and flash floods are all factors associated with climate change. An increase in the Earth's warming of up to 4.8 °C over the coming decades will, depending on the climate zone, result in an increase in heavy rainfall and storms contributing to an increase in the frequency and magnitude of flood incidents, but the increase in global temperature will also cause heat waves and droughts. These phenomena are already influencing the occurrence of nutrient deficiencies in plants, which may reduce crop yields and in the future may have significant impact on the economy, social development, geopolitics, local and national politics, law and health care [1,2]. Adverse phenomena are expected to worsen in the coming decades with increased variability and frequency of extreme droughts and uncertainty in rainfall distribution [3].

Currently, arid and semi-arid regions cover almost a third of the Earth's surface [4], and one of the observed effects of climate change is prolonged periods of drought. Drought is defined as a climatic condition with below-average or no rainfall for a long period of time. It leads to serious environmental, and also socio-economic, problems such as crop failure, water scarcity and food insecurity in a given area [2]. When drought occurs in a region that has previously received a lot of rainfall, plants become more sensitive to changes in water availability than species growing in arid or semi-arid regions. Therefore, drought can be described as a situation in which plants are unable to absorb water from the soil and are under water stress.

Long-term drought can cause desertification, degradation and soil salinisation, which significantly affect the soil ecosystem [5,6]; therefore it is necessary to look for solutions that increase the efficiency of water use, e.g., in agriculture [7]. Organic mulching, water fertilisation and the use of synthetic polymers are just some of the methods suggested in the literature for effective moisture management [7,8].

Superabsorbent polymers are very high molecular mass, crosslinked polyelectrolytes that can absorb more than 10 times their mass of water or aqueous solutions [9]. They are produced in the form of xerogel, i.e., a polymer with low moisture content in the form of granules [8] or fibres [10]. When added to water, these materials swell strongly, absorbing anywhere from several dozen grams to 1 kg of water per 1 g of starting polymer [11].

These materials have numerous practical applications [12] and are used to produce hygiene materials [13], such as disposable diapers, sanitary pads, dressings [14], slow-release drugs [15], and products for the immobilisation of microorganisms [16,17]; sealing telecommunications cables [9]; and in agriculture, horticulture, forestry and recultivation of degraded areas, substances increasing the water capacity of soil substrates, carriers of slow-release fertilisers or plant protection products [8,18,19]. SAP materials can be added directly to the soil, injected as a hydrated gel or dosed along with seeds during hydroseeding [20].

SAP materials have been continuously developed and refined for several decades. Classical polymers based on crosslinked acrylic copolymers [3], which are produced through petroleum processing, are increasingly being replaced by raw materials of natural origin [21–24].

SAPs are materials considered non-toxic and environmentally safe, but are produced mainly from vinyl monomers obtained from crude oil, natural gas and coal. Growing ecological awareness and taking actions to reduce the consumption of fossil fuels have significantly changed the perception of these materials and influenced new directions of research in this area. Numerous works are devoted to the chemical modification of biopolymers in order to increase their water absorption capacity. In many cases, these materials are cheap and easily available (cellulose, starch, chitin) and enable the use of waste products. Another strategy is the possibility of producing ecological, "green", acrylic monomers, mainly acrylic acid, from waste raw materials containing glycerol (byproduct obtained during the synthesis of biodiesel from vegetable oils), carbohydrates or organic acids. This solution opens a new path in the synthesis of very well-researched acrylic polymers from renewable raw materials. In this paper, databases are reviewed with regard to the structure, classification and methods of obtaining superabsorbent polymers, their use in agriculture and the remediation of degraded land, their effect on the microbial activity of soils and the factors affecting their degradation in soils. Particular focus is given to polymers based on acrylic acid acrylamide and their copolymers and their impact on the soil environment.

2. Polymers: Structure and Preparation

2.1. Chemical Composition of SAP

Superabsorbents, depending on their chemical composition, can be divided into two groups. The raw materials for production of acrylic SAP are mostly petroleum-based, relatively cheap synthetic copolymers produced from vinyl monomers and chemically modified natural polymers [25,26] based on starch [27–29], cellulose [30–34], chitosan [13,35–37], humic acid [38], proteins [39–41], alginate [42–45] agar [46], curdlan [47–49], gellan [50], xanthan [51,52] or arabic gum [46,53,54]. It is also possible to produce SAPs as a result of alkaline hydrolysis of polyacrylonitrile waste used for the production of fibres and fabrics [55] or a combination of several processes [56]. Many materials have also been created consisting of acrylic and/or natural polymers, both chemically modified and grafted polymers additionally containing other functional ingredients (Figure 1). Additions of mineral substances such as silica [57,58], attapulgite [59], bentonite [51–61] or kaolin [62] are often used.
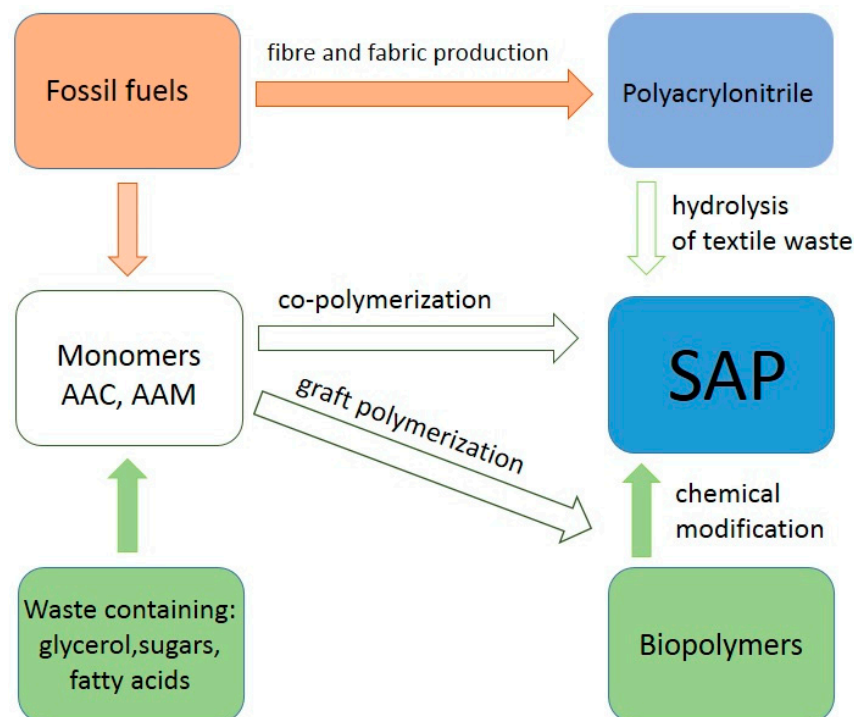
Figure 1. Raw materials and strategies used to produce SAP materials. Waste raw materials and natural materials that can be used to reduce the consumption of fossil fuels are marked in green (green path).

Depending on the properties of the modified biopolymer, there are different strategies to transform natural polymers into SAP materials. The main goal is to increase the ability to absorb water, i.e., to increase the number of groups capable of dissociation and functional groups forming hydrogen bonds. This method is used to modify, for example, cellulose into soluble carboxymethylcellulose, which is then transformed into an insoluble SAP polymer using crosslinkers such as polyhydric alcohols [63] or 1,4-butanediol diglycidyl ether [64].

Water-soluble hydrocolloids can be converted into insoluble gels by various substances. In the case of sodium alginate, difunctional amines [45] or aldehydes, e.g., glutaradehyde [65], are used.

A slightly different strategy for modifying biopolymers involves graft polymerisation. This process involves the polymerisation of vinyl monomers in the presence of biopolymers, e.g., starch [66,67], chitosan [35,36] or guar gum [68]. As a result of radical reactions, vinyl monomers are attached to the chains of natural polymers, creating branches in the form of hydrophilic polyelectrolyte chains [27]. Producing SAPs from sustainable biomass is of vital importance for avoiding CO_2 emissions and global warming.

2.2. Vinyl Monomers

Synthetic superabsorbents are obtained by polymerisation of vinyl monomers. From a chemical point of view, these are usually copolymers of, e.g., acrylic acid and other compounds containing one double bond with a crosslinker, which is a vinyl compound, containing more than one double bond [69].

Derivatives of acrylic acid and other vinyl compounds are used to synthesize superabsorbents. Acrylic polymers are produced by radical polymerisation of acrylic acid (AAC) [31], methacrylic acid (MAA) [70], 2-acrylamido-2-methylpropanesulphonic acid (AMPS) [71], acrylamide (AAM), N-isopropylacrylamide (NIPAM) [72], acrylonitrile (ACN) [73] or their mixtures (Figure 2) [70]. SAPs can be neutral, anionic or cationic. These polymers exhibit hydrophilic properties due to the presence of functional groups such as hydroxyl (–OH), carboxyl (–COOH), amide (–CONH–) or sulphone (–SO_3H) [70] groups.
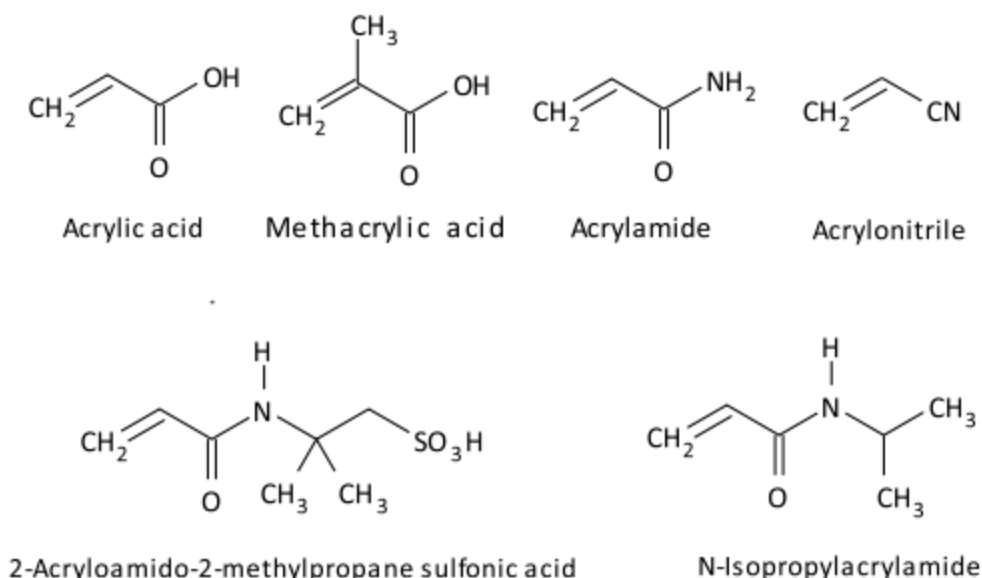
Figure 2. Vinyl monomers used in the production of SAPs.

Polymers obtained as a result of the polymerisation of acidic or alkaline monomers, i.e., those containing groups capable of dissociation, are classified as polyelectrolytes [74]. In the case of polymerisation of non-ionic monomers such as acrylamide or acrylonitrile, the obtained polymers can be transformed into polyelectrolytes as a result of hydrolysis of the amide [75,76] or nitrile group [73]. This process may occur before, during or after polymerisation as a result of reaction with a strong (sodium or potassium) base. Hydrolysis leads to the formation of acrylic acid, or rather its salt, i.e., alkali metal acrylate (Figure 3).
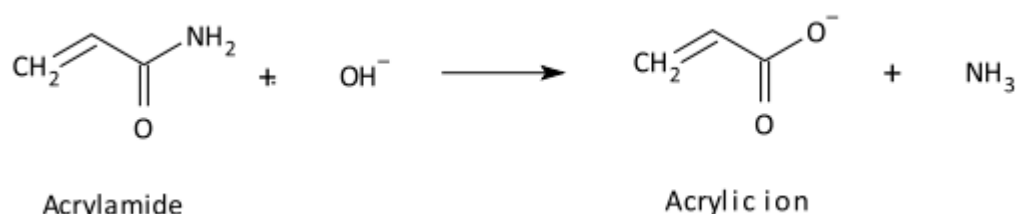

Figure 3. Hydrolysis of acrylamide in an alkaline medium.

The degree of neutralisation of acrylic (Figure 4) and methacrylic acid in the polymerized mixture varies. In the case of polymerisation of an acrylic acid solution, the highest absorbency is obtained after neutralizing the acid in the range of 60 to 85% mol, using a solution of sodium (potassium or ammonium) hydroxide and/or carbonate [35,70,77,78].

Figure 4. The reaction of neutralizing acrylic acid with a base.

Vinyl monomers are produced industrially from acetylene, propylene or propane, obtained from the processing of natural gas and crude oil [79]. Numerous works are being carried out on the synthesis of acrylic monomers from waste products containing glycerin [80], sugars [81], fatty acids [82] or the byproducts of waste lignocellulosic biomass [83]. In the first stage, 3-hydroxypropionic acid (3-HP) is produced microbiologically, from

which acrylic acid is then obtained as a result of catalytic dehydration (Figure 5). The disadvantage of microbiological methods is their relatively low biotransformation efficiency, low concentration of the resulting product and the related relatively high costs and large amount of waste generated during the concentration and purification of 3-HP. Acrylic acid can also be obtained by microbiological oxidation of malic or fumaric acid produced from organic waste materials [84]. The production of acrylic acid using biotechnological methods seems extremely promising. The advantage of SAP synthesis from sustainably obtained vinyl monomers is that further polymer synthesis is waste-free and, extremely importantly, these compounds can be used for graft polymerisation with biopolymers.
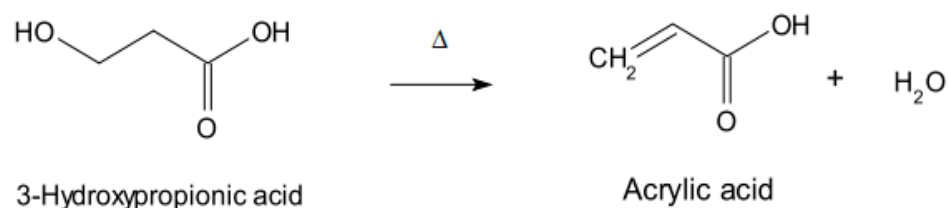

Figure 5. Thermal decomposition of 3-hydroxypropionic acid.

In order to prevent polymerisation during storage and transport, polymerisation inhibitors are added to acrylic acid and other vinyl monomers. Manufacturers often add, for example, hydroquinone monomethyl ether (4-methoxyphenol, MEHQ) to glacial acrylic acid in an amount of approximately 200 mg/kg [67,85]. A monomer that is to be stored for a long time is supplemented with larger amounts of MEHQ (Figure 6). This compound captures free radicals, and its action is particularly effective in the presence of molecular oxygen dissolved in acid. Oxidation of acrylic acid is a beneficial factor preventing polymerisation of the monomer during storage. MEHQ oxidation reactions using a thermal initiator such as persulphate are responsible for the colour of the finished product [86].
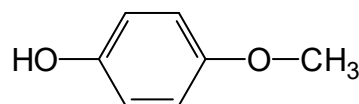

Figure 6. Hydroquinone monomethyl ether.

Monomer producers may also add other substances that stabilize free radicals (polymerisation inhibitors), such as phenothiazine (0.1%) [87,88], hydroquinone monomethyl ether (0.1%) [87], methylene blue (0.5–1%) and N,N′-diphenyl-p-phenylenediamine (0.05%) [89]. Stabilisers and oxygen dissolved in the acrylic acid prevent polymerisation during transport and storage, but in their presence, unfavourable non-radical processes occur [89,90]. One such transformation is the acid dimerisation reaction. As a result of the Michael reaction, an adduct is formed from two molecules of acrylic acid, 3-acryloxopropionic acid (β-acryloxypropionic acid, AOP) (Figure 7). The reaction can be repeated to form more complex polyesters (Figure 8) [91].
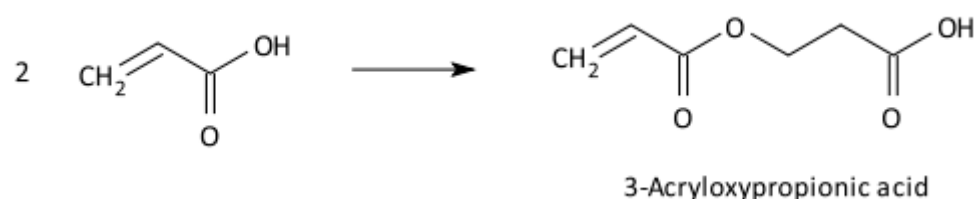

Figure 7. The formation of 3-acryloxopropionic acid as a result of the Michael reaction [92].
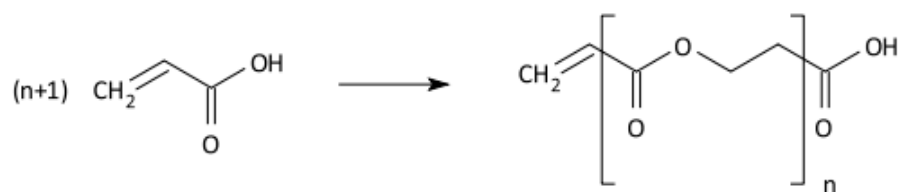
Figure 8. Linear polyester compounds formed from acrylic acid during the Michael reaction.

2.3. Radical Polymerisation of Vinyl Monomers

The initiation of radical polymerisation can be carried out in several different ways: the use of chemical redox initiators, UV radiation [50,93,94] or ionisation radiation [33]. In the first case, a pair of oxidant-reducer compounds is used to generate free radicals. For example, peroxides are used as oxidants: sodium persulphate (potassium or ammonium) (Figure 9), cerium ammonium nitrate [27], benzoyl peroxide [31] or aliphatic azo- compounds like 2,2′-azobisisobutyronitrile (AIBN) [70]. However, the role of a reducing agent can be played by sodium (potassium) metabisulphite, sodium bisulphate (IV), ascorbic acid, tetramethylethylenediamine (TMEDA) or ethanolamine [95–97]. Free radicals can be generated using the Fenton or Fenton-like reaction, in which the oxidant is hydrogen peroxide in the presence of Fe^{2+} or Cu^+, Co^{3+}, Ce^{3+} ions [98,99]. The emerging hydroxyl radical hydroxyl ($OH^\bullet$) or hydroxyl radical ($HO_2^\bullet$) initiates the polymerisation reaction of vinyl monomers (Figure 10).

$$S_2O_8^{2-} \rightarrow 2\ SO_4^{-\bullet}$$
$$SO_4^{-\bullet} + H_2O \rightarrow SO_4^{2-} + OH^\bullet + H^+$$

Figure 9. Generation of free radicals using persulphate [75,100].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^\bullet$$
$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2^\bullet + H^+$$

Figure 10. Fenton reaction [101].

When polymerisation is initiated using UV (UV photopolymerisation), photosensitisers, such as 2,2-dimethoxy-2-phenylacetophenone (Irgacure 651) (BDK) [34,102–104], 1-hydroxycyclohexyl phenyl ketone [105], 4,4′-bi(dimethylamino)benzophenone (MK) [60] or 2-hydroxy-2-methylpropiophenone [106], are added to the monomers (Figure 11).

2,2-Dimethoxy-2-phenylacetophenone

2-Hydroxy-2-methylpropiophenone

4,4'-Bi(dimethylamino)benzophenone

Hydroxycyclohexylphenyl ketone

Figure 11. Photoinitiators used in polymerisation initiated by ultraviolet light.

Polymerisation of monofunctional acrylic monomers leads to unbranched (linear) water-soluble polymers (Figure 12). These substances have numerous applications as thickeners, coagulants and stabilisers. In aqueous solutions, polyelectrolytes react with multivalent ions, combining various fragments of the polyelectrolyte, increasing the molar mass and reducing solubility [107]. To obtain water-insoluble SAP poly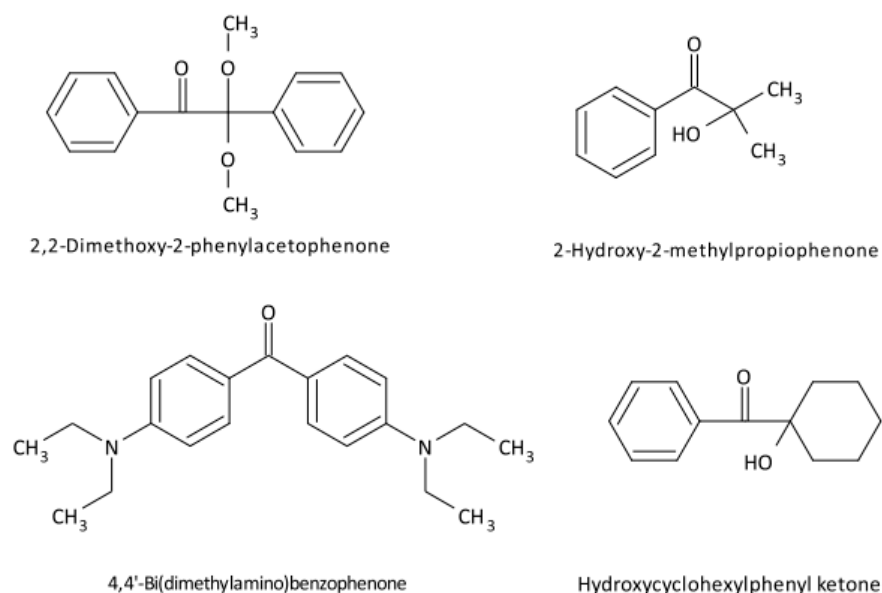mers, multifunctional vinyl monomers are used as the so-called crosslinker. These compounds copolymerize with acrylic monomers, creating a multidimensional network. The crosslinkers are added before polymerisation in concentrations of approximately 500 ppm to 1% (0.01–0.2% mol) in relation to the acrylic acid. Its amount is selected so that the crosslinking reaction product has the desired absorbency. Crosslinkers may contain 2, 3 or 4 vinyl groups (Figure 13). The more double bonds, the more linear chains are attached to one node of the polymer network. The following crosslinkers are used in the production of SAP: N,N′-methylenebis(acrylamide) (NMBA), triethylene glycol dimethacrylate (TEGDMA) [108], ethylene glycol dimethacrylate (EGDMA) [109,110], glycerol acrylate (GA) [99], 1,4-butanediol diacrylate (BDDA) [111], butylenes dimethacrylate (BDMA) [70], ethylene glycol diacrylate (EGDA), allyl methacrylate, allyl acrylate, diethylene glycol diacrylate (DEGDA) or trimethylol propane triacrylate (TMPTA) [112].
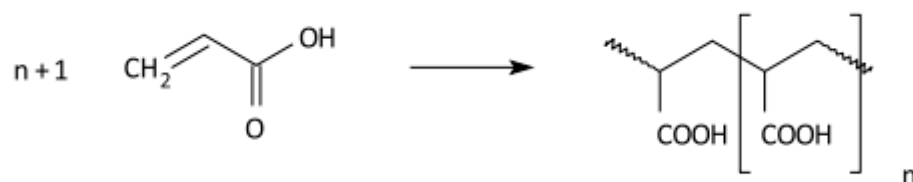

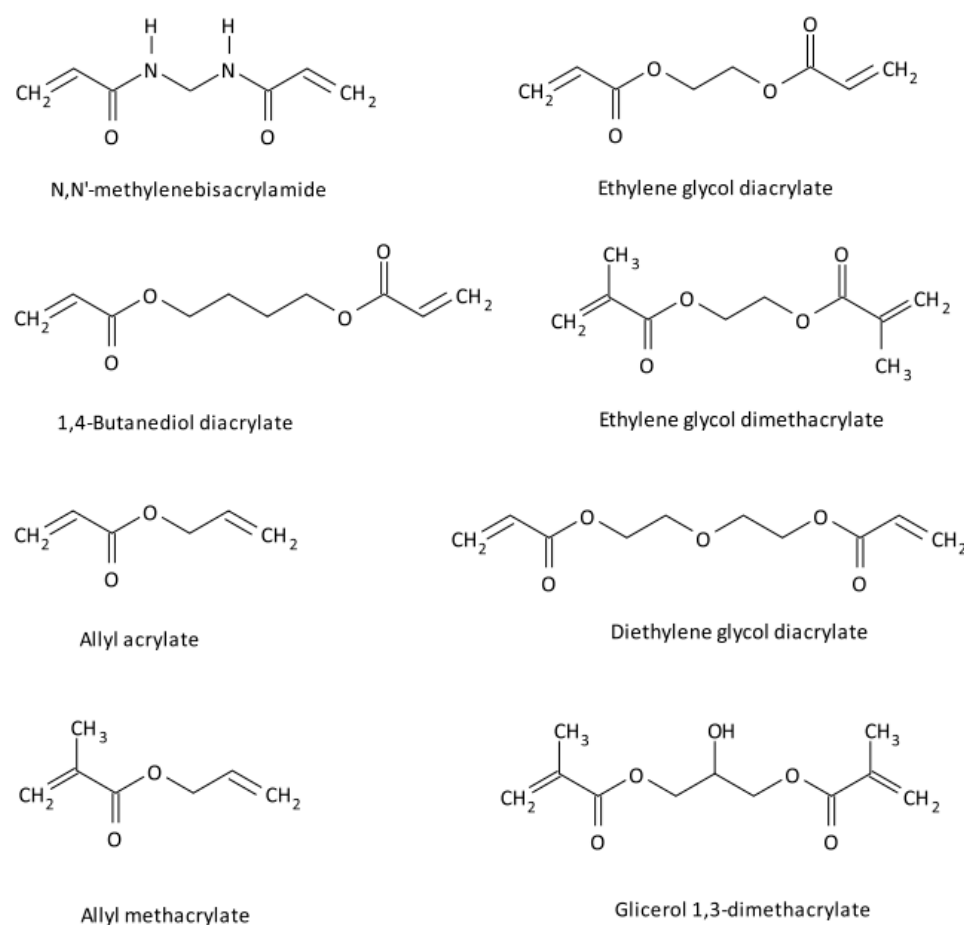**Figure 12.** Polymerisation of acrylic acid.

Figure 13. Bifunctional crosslinkers used in copolymerisation with vinyl monomers.

Before polymerisation, chelating [113] and dispersing compounds and surfactants are also added to help dissolve hydrophobic crosslinkers. Polymerisation can be carried out in solution or bulk polymerisation [69] and suspension polymerisation [3,114]. In the case of modification of natural polymers, it is possible to graft polymerized vinyl monomers onto starch, cellulose and other natural polymers, such as guar gum [68]; alginate [43,44,115,116] or chemically modified biopolymers, e.g., carboxymethylcellulose [117]; or oxidized starch [118]. In the case of superabsorbents containing starch or polyvinyl alcohol (PVA), crosslinking substances containing two aldehyde groups are additionally used, namely glutaraldehyde and di- and tricarboxylic acids: succinic, malonic or citric [29].

During polymerisation, 3-acryloxopropionic acid reacts with acrylic acid molecules and can be incorporated into the polymer network (Figure 14) [119].
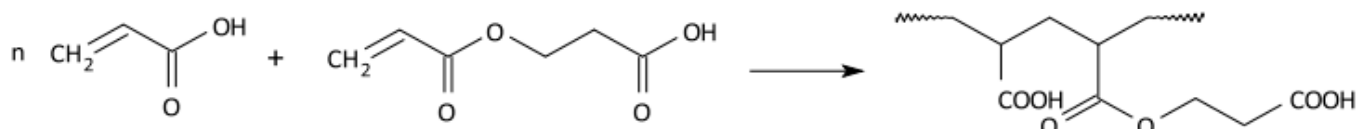
Figure 14. Incorporation of 3-acryloxopropionic into the polymer chain [119].

Drying and heating the polymer containing built-in dimer molecules leads to hydrolysis and the release of unbound acrylic acid as a result of a process opposite to the Michael reaction [119]. The speed of this reaction (Figure 15) depends on the temperature. During the production of an SAP, alkaline hydrolysis of the dimer is possible before polymerisation, resulting in the formation of 3-hydroxypropionate in addition to acrylic ions (Figure 16) [119].

Figure 15. Decomposition of 3-acryloxopropionic molecules incorporated in the polymer chain as a result of heating the polymer.

3-Acryloxypropionic ion   Acrylic ion   3-Hydroxypropionic ion

Figure 16. Alkaline hydrolysis of acrylic acid dimer [119].

Most of the 3-HPA produced during hydrolysis passes through the polymer drying stage and remains in the finished product. However, drying should be carried out at relatively low temperatures, because strongly heated (175–200 °C) 3-hydroxypropionic acid decomposes with the release of acrylic acid (Figure 5). This reaction increases the content of free acrylic acid in the finished product to the values obtained without the hydrolysis step [119].

2.4. Chemical Properties of SAPs

From a chemical point of view, the vast majority of SAPs can be classified as polyelectrolytes [120]. These compounds, although insoluble in water, dissociate in this solvent. Acrylic polymers are cation exchangers that can exchange cations with the environment. As

a result of these processes, cations, e.g., potassium or ammonium cations, can be introduced into the environment in a controlled manner, which, due to their excellent solubility, are otherwise quickly washed away by water (Figure 17). Reactions of this type also increase the buffer capacity of the system. Hydrogen, potassium or sodium ions contained in SAPs are released into the environment and replaced by other monovalent or multivalent ions (Figure 18), e.g., calcium, magnesium [121] and heavy metals [122]. The binding of multivalent metal ions involves an increase in the degree of crosslinking of the polymer (Figure 19), which reduces water absorbency and causes the gel to shrink [123]. In an acidic environment, the reverse reaction may also occur—the equilibria (Figure 18) shift to the left, which may result in the release of multivalent ions into the environment and the degree of crosslinking of the polymer may decrease (Figure 19). The gel containing fertiliser ingredients also slows down their extraction due to diffusion resistance. Changing the degree of crosslinking directly affects the mass transfer through the gel layer. The crosslinking occurring with multivalent metal ions reduces the release rate of agrochemicals and other water-soluble substances contained in SAPs.

Figure 17. Release of potassium cations by ion exchange from poly(potassium acrylate).

Figure 18. Binding of multivalent ions (Me^{2+}) by poly(acrylic acid) molecules.

Figure 19. Binding of divalent ions (Me^{2+}) by poly(acrylic acid) molecules combined with increased crosslinking of the polymer.

3. Agricultural and Degraded Land Reclamation Applications

3.1. Agricultural Application

Research into the use of polymers as soil conditioners has been carried out since the 1950s [124]. Since the 1990s, there has been a growing interest in polymers as additives to

soils and degraded soils to protect them from erosion [125,126], improve the hydrophysical properties of arable land [127–129], reverse desertification [130,131] and retain soil moisture [126,132].

Acrylic polymers can also help grow crops in saline areas. The observed climate changes contribute not only to water shortages but also to increased soil salinity. It has been shown that the addition of a composite containing an SAP with commercial biochar to soil increases the resistance of Quinoa (*Chenopodium quinoa* Willd.) to salinity [133].

SAP polymers used as soil additives, due to their availability in price and stability in the soil environment, are usually based on crosslinked polyacrylates or polyacrylamides and, due to their specific properties and high absorbency of aqueous solutions, can be used as soil conditioners [8,11].

3.1.1. Influence of SAPs on Soil Structure and Soil Water Retention

Water in SAP hydrogels is mainly bound by hydrogen bonds. In the case of porous and fibrous polymers, a small part of the free water is found in the microcapillaries of the gel (Figure 20). The soft hydrogel changes the structure of the soil and, in particularly permeable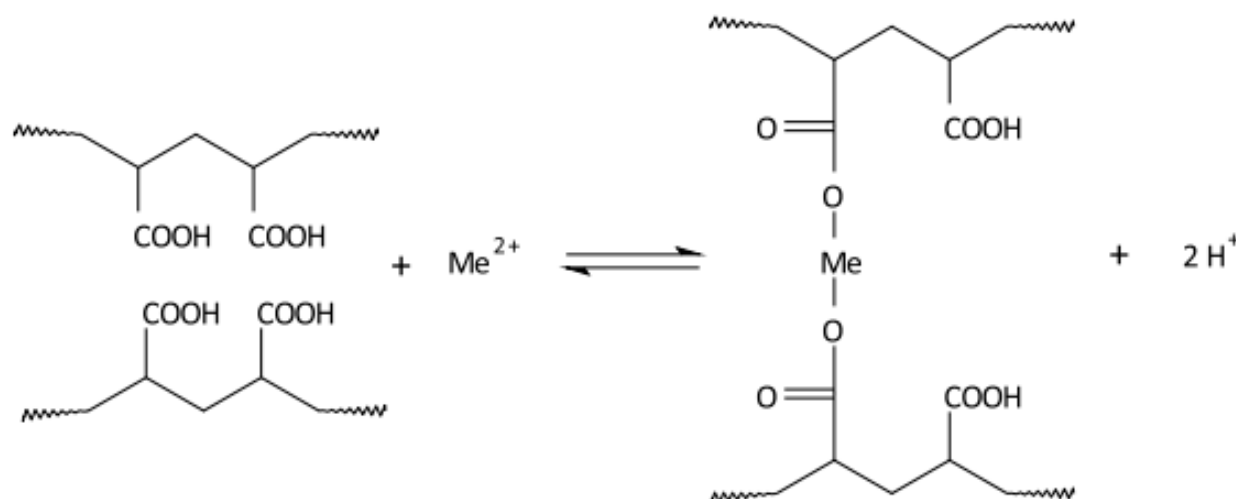 sandy substrates, blocks soil capillaries, thus also increasing water retention. Water vapor pressure over the hydrogel is much lower than over pure water. In the gel, water is strongly hydrogen-bonded to the polymer molecules. The hydrogen atoms of water molecules interact with the oxygen atoms in carboxyl and hydroxyl groups or nitrogen atoms in amide groups. Such bound water evaporates slowly, even during periods of drought.

Figure 20. Porous sap hydrogel containing hydrogen-bonded water and free water in capillaries.

Extensive research on the effects of SAPs on soil structure was conducted by Paluszek [134–136]. The use of acrylic polymers as an additive to the eroded soil significantly not only increased water absorption, but also influenced total porosity and the number of macropores > 20 μm and air permeability. The application of polyarylamide increased the proportion of the 1–2 mm fraction [137].

Zhao's study found that different SAP contents had a significant effect on soil particle size distribution [138]. Due to the strong water absorption properties of SAPs, SAP-treated soil changed the size distribution of soil particles, and thus the soil structure also changed. The greatest influence of added polymers was observed for the content of clay particles [139].

The most important property of superabsorbent polymers, from the point of view of climate change and emerging long periods of drought, is their ability to absorb large amounts of water. The use of synthetic superabsorbent polymers is an alternative to improving water retention and availability for crops in water-starved production systems [140–143]. SAPs have been successfully used to reduce water stress in a wide variety of crops, including citrus, maize, barley, rapeseed, tomatoes, grapevines and many others [144–149], because the water absorbed by SAPs is available to plants. The impact of SAPs was also studied in a number of tree species, including pine [150,151], *Eucalyptus grandis*, *Eucalyptus citriodora*, *Pinus caribaea*, *Araucaria cunninghamii*, *Melia volkensii*, *Azadirachta indica*, *Maesopsis eminii*, *Terminalia superba*, *Picea abies* and *Fagus sylvatica*. Not only was an increased availability

of water observed, but so was a significant reduction in tree transpiration compared to control soils without SAP addition [152]. Reduction in evapotranspiration was reported by Agaba [153]. It is probable that soil amendment with SAPs decreased the hydraulic soil conductivity that might reduce plant transpiration and soil evaporation. In their study, Zhao et al. proved that soil mixed with SAPs significantly reduced soil water evaporation. An SAP concentration of 0.2% can reduce soil water evaporation enough to meet plant growth needs [138].

It has been estimated that around 90% of the water absorbed by SAPs is available to plants [154]. SAPs do not decrease vertical water flow through soil profile and do decrease leaching of nutrients from soils. The advantages of the amendment of soils with SAPs include increased plant available water in soils, faster growth of plants and prolonged survival of plants under water stress [138,155].

Benefits of modifying soils with SAPs include increased water availability to plants in the soil, faster plant growth and longer plant survival under water stress conditions and reduced water evaporation rates [142,155–157].

A swollen hydrogel acts as a local miniature water reservoir, which helps with initial crop establishment and results in better plant growth [157]. Singh et al. found that higher soil moisture retention when using SAPs and then its gradual release over a longer period of time allows the plant to better use moisture in the root zone with less frequent irrigation [158].

Similar conclusions were reached by Demitri et al., who investigated the possibility of using cellulose-based hydrogels as a carrier for long-term and controlled water release in arid and desert areas. According to the authors, the main advantage of hydrogels is that they can control the release of stored water as the soil dries out, maintaining soil moisture for a relatively long time. In addition, SAPs increase soil porosity, providing better oxygenation for plant roots [156].

The size of the SAP fraction also affects water retention and availability for plants. Smaller gel particles can block more soil capillaries, reducing the permeability of the substrate and retaining water in the spaces formed between the swollen hydrogel particles [109,159].

In summary, the use of SAPs extends the inter-period irrigation period, and the time to achieve soil dryness is delayed [141], thus reducing the amount of water used in irrigation, the frequency of irrigation and the total water demand of the crop [157,158]. SAP application improves the physical properties of the soil, significantly increasing field capacity moisture content, plant-available water content and relative field capacity, retention pores, water-stable structural units and structural coefficient and reduced transmission pores, penetration resistance and saturated hydraulic conductivity [141].

3.1.2. Fertiliser Release into the Soil by SAPs

SAPs can be saturated with fertiliser compounds (e.g., soluble phosphates, potassium ions, nitrogen compounds) or plant protection products, which are not immediately leached by water, but are gradually released into the soil environment and then absorbed by plants (Figure 21) [21,156,160]. The reduction of nutrient leaching by superabsorbents is achieved in two ways: by absorbing fertiliser compounds and by retaining water in the soil, so that superabsorbents prevent soluble compounds and colloids from leaching into deeper soil layers and draining into watercourses. The rate of diffusion of substances contained in the hydrogel, both fertiliser ingredients and agrochemicals, also depends on the size of the individual SAP particles, the degree of crosslinking of the polymer and its macroscopic structure.
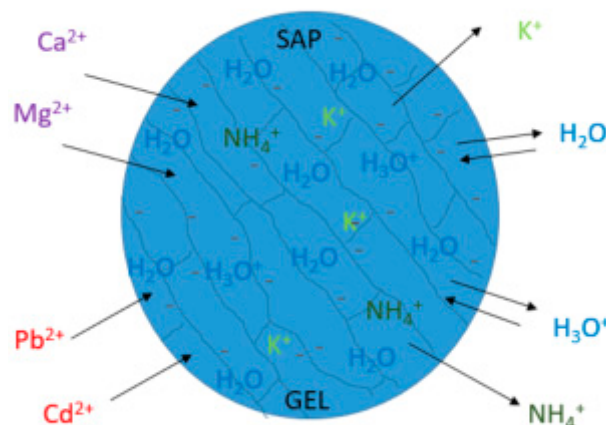
Figure 21. SAP gel made of anionic polyelectrolyte, e.g., potassium-ammonium polyacrylate. Negative charges are located on the polymer chains, contributing to the repulsion of the chains; water is bound by hydrogen bonds with carboxyl anions and remains in a state of thermodynamic equilibrium with the environment, absorbing and releasing water. The gel stabilises the pH, i.e., the concentration of oxonium ions (H_3O^+); as a result of dissociation and diffusion, it slowly releases potassium and ammonium ions and absorbs polyvalent ions, which contribute to the shrinkage of the gel.

Just as water absorption by SAPs is caused by differences in water activity between the interior of the gel and the exterior fluid, fertilisers or agrochemicals that are initially of higher activity in the polymer will diffuse out of the particles into the surroundings. The initially dry polymer material (xerogel) is activated to release the substance by swelling. The factors influencing this process depend on the change in the diffusion capacity of the released substance [160]. The swelling of poly(acrylic acid) and other anionic and cationic polymers depends on pH, ionic strength and the concentration of polyvalent ions (Figure 21). These factors can act as parameters controlling the release of substances. The crosslinking density also influences the degree of swelling and is used to control the substance release process. In order to create a polymer particle characterised by controlled release, typically the polymer is obtained first and then the active substance is added to the polymer by saturating/swelling the finished polymer in a solution of the active substance and then drying the gel [18,160–163].

SAPs increase retention and prevent leaching of pesticides from the soil [164]. A variety of polysaccharides such as chitosan, pectin and carboxymethylcellulose have been used to prepare hydrogels as a soil-stimulating fertiliser release system [24,143].

3.2. Reclamation

Studies on the application of superabsorbents in degraded soils are less common than those in arable soils and mostly describe the effect of SAP application on plant performance. SAPs increase the amount of available water in the soil, allowing plants to survive longer under water stress conditions. Their addition to the soil reduces the evapotranspiration rate of plants. They induce significantly higher growth rates in plants growing on SAP-modified soil. Research carried out using SAPs for remediated and degraded land focuses mainly on their effect on the metal content of the soil. They bind heavy metals and reduce their negative effects on plants [152,165].

Zaho [166] has shown that acrylamide/acrylic acid cellulose hydrogels that SAPs absorbed Cu (II), Pb (II) and Cd (II) metal ions from solutions under different conditions (Figures 18 and 19).

In his research, Torres tested whether a Na-K polyacrylate polymer could be used to remediate sandy soil artificially contaminated with copper. The copper content of the soil with SAPs decreased and the polymer particles removed from the soil contained high levels of copper [167].

Qu G et al. investigated the use of polyacrylate polymers to aid thephytostabilisation of mine soils. In a pot study, they found that the application of the polymer led to an increase in the enzymatic activity of the soil. Water-extractable Cu ad Zn levels of soil decreased after polymer application [168].

Guiwei et al. investigated the effects of different doses of polyacrylate polymers on plant growth and mine soil quality from Pb-contaminated. The polymer increased the water-holding capacity of the soil but this capacity decreased over time. Application of polyacrylate polymers reduced bioavailable Pb from 15 to 66% of those in the unamended soil, depending on the polymer application rate [169].

3.3. The Influence of Superabsorbents on the Microbiological Activity of Soils

Soil microorganisms are extremely important for the proper functioning of terrestrial ecosystems, participating in key processes such as energy flow, nutrient cycling and organic matter turnover in terrestrial ecosystems, humification processes, contaminant degradation and maintenance of soil structure [170]. Microbial activity has a direct impact on the functioning of ecosystems and is directly related to the quality of soils; the rate of mineralisation of soil organic matter may depend on the composition of microbial communities and their ability to metabolise various organic compounds [171].

Soil environment, human industrial activities and cultivation practices affect microbial activity in soils [170–172]. Consequently, altering the microbiome may affect plant productivity and diversity [173,174].

The soil microbial biomass plays an important role in the biogeochemical cycling of soil nutrients, through the production of various enzymes involved in mineralisation and immobilisation processes, and thus plays an important role in maintaining soil health [175]. One method of studying soil microorganisms is to determine the enzymatic activity of soils [170,176,177]. Enzymes are products of the activity of microorganisms. They are proteins that act as catalysts in many biochemical processes in the soil associated with the cycling of carbon, nitrogen, phosphorus, sulphur and other nutrients [170,172]. Therefore, determining the activity of soil enzymes can provide information on the cycling of these elements in the soil and their availability to plants. Soil enzyme determinations are often used to assess the condition of reclaimed soils [178–180] However, interpretation of the results obtained is difficult, as the measured activity of soil enzymes is influenced by a number of factors that sometimes act antagonistically [181]. The enzymatic activity of soils is influenced by a number of other factors, including abiotic factors such as temperature, humidity the granulometric composition of the soil [181] Sandy soils have low enzymatic activity, clay soils have average activity, and humus soils have high enzymatic activity [182]. Soil enzymes are not only produced by microorganisms, but can be products of plant root secretions or be of animal origin [183]. In addition, extracellular enzymes such as urease or phosphatases can accumulate in clay or organic material and show false results of soil microbial activity [176,183].

The addition of SAPs to the soil could be beneficial to soil microorganisms, as they could use SAPs as a source of carbon and/or nitrogen [184,185].

The results of studies to assess the impact of SAPs on soil microbial communities are inconclusive and sometimes contradictory. For instance, Kay-Shoemake et al. [184] reported larger populations of heterotrophic culturable bacteria in polyacrylamide-treated soils planted to potatoes but not in those planted to bean.

Sojka et al. [128] reported a negative effect of large doses of polyacrylamide superabsorbent on active microbial biomass but relatively weak effect on the CLPPs measured with Biolog® testing for arable soils in Idaho.

Awad et al. [186] studied arable soils in Korea and reported a weak effect of polyacrylamide, biopolymer and biochar application on soil enzyme activities for soils that did not receive plant residues, but a strong stimulating effect was observed for soils that received plant residues and were treated with biopolymer and biochar.

Similar results were obtained by Bana et al. [7]. Dehydrogenase, alkaline phosphatase, acid phosphatase and urease activities increased with the simultaneous application of crop-residue mulch and SAPs. This positive effect was also observed on soil microbial (bacteria, fungi, actinobacteria) populations [7].

Sojka et al. suggested that the effect of superabsorbents on soil microbial properties may depend upon crop cover, cultural practice and on soil properties (e.g., nutrient contents) [128].

Studies to evaluate the use of SAPs on the microbiological properties of post-industrial soils are rare and limited to mine soils contaminated with toxic metals [182,187]. These studies reported a positive effect of SAP application on soil microbiological properties [168,169,187–189], resulting from increased pH and significantly reduced concentrations of toxic metals in the soil solution.

3.4. New SAPs Environmentally Friendly and Plant-Friendly

Currently, research is being carried out to prepare low-cost and environmentally and plant-friendly SAPs.

Copolymerisation of natural polysaccharides, such as alginate [42], starch [161], cellulose [30], saccharose or chitosan [190–192] with hydrophilic synthetic polymers is one method of producing environmentally friendly polymers.

Polysaccharides and proteins are most commonly used in the preparation of SAPs due to the availability of raw material on an industrial scale and their biodegradability [11,30,104,152,193,194]. These copolymers can be degraded by natural biological processes, including the action of enzymes, microorganisms and water, transforming into harmless simple compounds that are safe for the environment [27,195].

With the addition of natural polysaccharides, the water absorption and mechanical strength of the copolymers are reduced [93,104,159,196].

Technologies for obtaining copolymers with starch, carboxymethylcellulose and natural additives of charcoal or bentonite are being investigated to increase the mechanical strength of the resulting SAPs [197,198].

Also, the use of only natural polymeric components such as chitin or chitosan in agricultural crops has an impact on increasing yields and agro-environmental sustainability [199,200]. Chitosan, as a biostimulant [201,202], improves plant tolerance to salinity [203], temperature [204] and drought [205] and promotes water uptake [202,206].

3.5. Restrictions on the Use of SAPs

However, there are limitations to the use of SAPs and factors that reduce their swelling capacity. Abiotic factors include salinity or the content of multivalent cations in the soil. SAP polymers contain ionic functional groups, such as carboxyl groups in polyacrylates, which reduce their ability to swell when multivalent metal ions are present in the soil and/or irrigation water. These cations form crosslinking bonds between carboxyl groups, contributing to a reduction in water uptake capacity during many drought and hydration cycles [207,208]. Zhou et al. reported that with higher cation valency, concentration and ionic radius, water uptake by polymers decreases significantly $Na^+ < K^+ < Mg^{2+} < Ca^{2+} < Fe^{2+} < Fe^{3+} < Al^{3+} < Cu^{2+}$ [209].

The high absorbency of SAP materials in demineralised water does not translate into high absorbency in water with higher ionic strength [121] and another important parameter, which is absorbency under load. The effectiveness of SAP application depends on the application depth and bulk density of the soil [210].

Another limitation in the use of hydrogels for agricultural applications is their low mechanical resistance [21]. The pressure exerted by the plant and the soil layer on the SAPs influence the loss of swelling capacity, elasticity and stiffness. To maintain polymer elasticity, long-chain molecules and adequate interbreeding are needed to dissipate external mechanical stresses on a hydrogel. The solution to the problem may be new methods of obtaining hydrogels with increased mechanical elasticity and chemical properties using

nanofibres [211]. The high surface area-to-volume ratio of nanoparticles and nanolayers has made them a good choice for use in polymer material networks [212]. Physical/chemical crosslinking of polymer chains with different structures at the micro/nano scale leads to networks with new unique properties (e.g., has significant water retention properties and can withstand large changes in pH, temperature and ionic strength of the swelling solution) [109].

There are also reports that have shown not only little or no response of hydrogels to crop yields [207,213], but also negative effects of hydrogels on root biomass and plant growth [214].

For agricultural applications, it has been shown that the optimum soil application rate of SAPs is 0.2% SAP per area or lower. If the superabsorbent polymer content is less than 0.2%, it improves the physical properties of the soil [215], increases the volume of the growing medium due to expansion after water absorption and expands the root growth space, which is beneficial to plant growth [216]. Higher doses may negatively affect the growth and development of plants and disturb root development and negatively affect root biomass [214,217]. The results of Situ's research showed that the use of superabsorbent polymers to eliminate the effect of lack of water resulted in damage to the roots and a reduction in their biomass [214]. Superabsorbent polymers form a three-dimensional network [153]. Plant roots can penetrate the hydrogel or attach to the hydrogel surface to gain direct contact with the water. Particularly in dry environments, roots accumulate around the hydrogel [218].

After applying higher doses of sodium and potassium acrylic polymer, the content of Na^+ and K^+ in the soil increased significantly. Excessive accumulation of Na^+ and K^+ ions resulted in a decrease in biomass, root length and root surface area and inhibited the uptake of some nutrients by plants [214]. In parallel, the ionic content has decreased Ca^{2+} and Mg^{2+} in soil and crops after application of superabsorbent polymers. Sodium polyacrylate had a more negative impact on crops than potassium polyacrylate [214].

It is extremely important to use SAP materials carefully and thoughtfully. Excessive amounts of xerogel added to the soil may cause adverse effects, including drying of plant roots. The added polymer absorbs water and reduces soil moisture [219]. The use of SAPs does not always produce noticeable results. If the soil moisture is optimal, the added polymer does not significantly affect plant growth [220].

Superabsorbents are, by definition, substances insoluble in water, but they contain numerous components that migrate into the solvent during the swelling of the xerogel [221]. In the case of these materials, we can talk about the soluble fraction consisting of unreacted monomer molecules, crosslinkers, necessary additives introduced before polymerisation, surfactants, solvents and low-molecular or non-crosslinked polymer fractions (Figure 13), as well as many other substances constituting both impurities of raw materials and by-products of reactions occurring during storage and polymerisation [86,119]. These substances significantly affect the quality, properties and possible applications of SAPs. This characteristic feature of superabsorbents seems to be underestimated or even ignored during their application or research. Substances migrating from the polymer change pH, ionic strength, surface tension, selectively bind ions limiting their availability in the environment, stabilise suspensions or flocculate and remove them [222–224]. Seemingly the same materials produced by different manufacturers may in practice differ dramatically in terms of the qualitative composition of the soluble fraction.

SAP polymers are considered non-toxic and harmless because they are substances insoluble in water, i.e., indigestible, even after entering the digestive tract of a human or animal. The above-mentioned substances eluted from polymers end up in the environment, and their actual impact on living organisms is currently unknown. Manufacturers often deliberately add various substances to SAP polymers to enrich the material with fertilizing substances, such as urea [45] or functional substances, e.g., humic acids [104]. In such cases, substrates introduced together with monomers before polymerisation increase the number of by-products and the concentration of soluble polymer fractions, which contributes to

the even greater complexity of the problem of migration of often unknown substances into the environment. The concentration of water-soluble components is in most cases small, but this problem requires further research. This is especially important for polymers introduced into the environment on a larger scale.

3.6. Degradation of SAPs in Soils

SAP degradation can result from various causes, such as chemical hydrolysis, mechanical or biological degradation, irreversible thermal degradation, photochemical oxidation and UV radiation [185,225,226].

The speed of these reactions depends on many factors such as chemical composition and the degree of crosslinking. Long carbon chains of polymers (C-C bounds) can be broken by radicals. UV radiation and oxidation processes occurring in the soil can generate radicals and reactive oxygen species [227,228]. Radicals break the polymer chains and cause the hydrated gel to break down and form colloidal solutions. Soil microorganisms, in particular fungi and certain strains of soil bacteria, are capable of degrading synthetic superabsorbents [185,229–231].
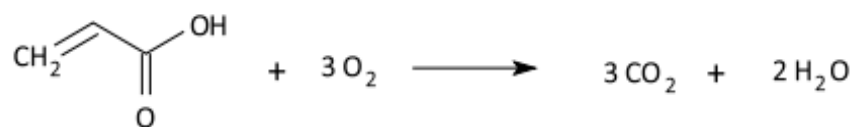
As a result of the degradation of synthetic polymers, soluble fractions of acrylic polymers are formed. Linear polyacrylates and polyacrylamides have been used for decades as additives to prevent erosion, stabilise soil, prevent leaching of colloidal particles from soil and clarify water [232,233]. Thus, it is likely that most of the soluble linear polymers present in SAPs (Figures 8 and 13) and the low-molecular-weight water-soluble polymers formed during the degradation of hydrogels will be adsorbed on the surface of soil colloids. The multivalent cations present in the soil environment form bonds between the negatively charged components of the soil and the anionic groups of the polymers. The degree of polymer adsorption depends on the type and size of the macromolecules, the ionic strength and concentration of the multivalent cations, the number of colloidal particles and the pH of the soil. Soluble polymer fractions may additionally contribute to changing the soil structure and may reduce both water and wind erosion [125,139].

Chemically unbound monomers present in SAPs, such as acrylic acid or acrylamide, are perfectly soluble in water. In the environment, they can easily get into the soil and groundwater. Due to the potentially toxic properties of acrylamide, special attention has been paid to this compound [234,235]. Acrylic acid derivatives are relatively quickly metabolised in the soil by microorganisms. Acrylamide is converted to ammonia (or ammonium ion) and acrylic acid (Figure 22), which can be decomposed through a series of biochemical reactions to water and carbon dioxide (Figure 23) [66]. Potentially, microorganisms in the soil can use acrylamide as a source of nitrogen [227,236,237] and acrylic acid as a source of carbon, because it can be metabolised in microbial cells, similarly to other low molecular weight alkyl acids.

Figure 22. Enzymatic decomposition of acrylamide in soil.

SAP degradation processes can be intensified by producing materials based on biopolymers or by creating copolymers with natural materials [238,239]. Natural materials based on carbohydrates or proteins undergo rapid enzymatic hydrolysis because their chains contain numerous glycosidic or peptide bonds [193]. Accelerating the decomposition of SAPs in the soil obviously has advantages and disadvantages. The rapid decomposition of the polymer may require more frequent application and increase costs, but at the same time it may significantly affect the development of soil microorganisms.

$$CH_2=CH-COOH + 3 O_2 \longrightarrow 3 CO_2 + 2 H_2O$$

Acrylic acid

Figure 23. Oxidation of acrylic acid.

Another still unsolved problem is the decomposition of polyacrylates under UV light and the need to cover the swollen polymer layer with a soil layer to counteract the premature decomposition of macromolecules due to radical reactions [184]. Some reports indicate that polymers degrade to monomers in the presence of light [240].

According to a study by Sarmah and Karak, starch-modified polyacrylic SAPs was degraded by 40% after three months after application to the soil [241]. In the case of cellulose-acrylic materials, the biodegradation process lasted only eight weeks [239].

4. Conclusions

Superabsorbents are materials that absorb huge amounts of water and are used for numerous applications that take advantage of this unique property of these materials. This article presents a review summarizing the latest research on the synthesis of polymeric superabsorbents (SAPs), their degradation in soil and their use in agriculture and for the remediation of degraded land. The review covers the use of various synthetic, natural, synthetic and combined materials as SAPs.

SAPs enable the introduction and enhancement of retention of water, minerals, fertilisers, crop protection ingredients and immobilised microorganisms in the soil. The slowed, programmed release of chemical ingredients from insoluble hydrophilic polymers allows for increased retention of these substances in the soil, but also reduces leaching of ingredients by groundwater and pollution of ground and surface water. A review of the articles shows that the dose of SAP applied to the soil is important. Too much SAPs can cause soil drying out of the application site and stunted plant root growth. SAPs influences positive changes in the substrate structure. Repeated cycles of swelling and shrinking of the gel loosen and aerate the soil.

Recent research concert the use of SAPs based on modified biopolymers and their copolymers with synthetic polymers (eco-friendly polymers). Such polymers are inexpensive to obtain (waste utilisation—the green path), increase yields and affect agro-environmental sustainability. However, it should be noted that the use of environmentally friendly SAPs is associated with relatively rapid hydrolysis of glycosidic or peptide bonds and decomposition of biomolecules by microorganisms, which accelerates the biodegradability of the polymer.

Due to the wide variety of chemical structures of modified biopolymers, their effects in the soil environment may vary. The production of synthetic SAPs is virtually waste-free, but when processing biopolymers hazardous waste products must also be taken into account. The development of biotechnological efficient production of acrylic acid may be a breakthrough in the sustainable production of SAP materials without the use of fossil fuels.

Superabsorbents materials contain numerous water soluble substances, such as polymerisation inhibitors and initiators, residues of unreacted monomers and by-products of oxidation and monomer transformations occurring during storage, polymerisation, hydrolysis and thermal degradation, crosslinkers, stabilisers, other excipients and soluble polymer fractions. These substances occur in SAP materials in small, sometimes even in trace amounts, but in an environment with limited water they can potentially significantly affect pH, ionic strength, viscosity and surface tension. These factors may significantly affect soil microorganisms. Basic analyses performed by manufacturers and controlled parameters, such as absorbency, absorbency under load and the amount of unreacted monomers are not fully sufficient to predict the processes occurring in the environment, especially the

impact on soil microorganisms. This issue requires more extensive research. It is possible that some of the research results and sometimes contradictory conclusions from specific SAP applications are the result of significant differences in the chemical composition of commercially available materials.

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