

Article

Experimental Assessment of the Use of a Novel Superabsorbent polymer (SAP) for the Optimization of Water Consumption in Agricultural Irrigation Process

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Abstract: In this work, an innovative cellulose-based superabsorbent polymer (SAP) was experimentally assessed as an environmentally friendly alternative to acrylate-based SAPs, for the optimization of water consumption in agriculture. The cellulose-based SAP was synthesized and tested for its swelling capability in different aqueous media. The effectiveness of the SAP in agricultural applications was then evaluated by analyzing its performance after several absorption/desorption cycles, over a period of approximately 80 days, upon addition to different types of soil, *i.e.*, white and red soil, for the cultivation of two varieties of plants typical of the Mediterranean area (tomatoes and chicory). The results confirmed that SAP-amended soil can store a considerable amount of water and can release it gradually to the plant roots when needed. The adoption of the proposed SAP in cultivations could thus represent a promising solution for the rationalization of water resources, especially in desert areas.

Keywords: agriculture; hydrogel; irrigation optimization; superabsorbent polymer; water management

1. Introduction

The optimization of the use of water resources is strategic for the long-term competitiveness of the agricultural industry. Indeed, water shortage has become a serious issue, especially in those areas (such as some areas of the Mediterranean Basin) that are exposed to the progressive desertification. On the other hand, in coastal areas, saltwater intrusion (*i.e.*, the movement of saline water into freshwater aquifers [1]), may lead to an increase of the salinity of water, thus making it unusable for irrigation. Water management is considered one of the major challenges of the near future [2]; in fact, by 2030, water demand is expected to be 50% higher than today, and withdrawals could exceed natural renewal by over 60%, resulting in water scarcity [3].

In such a context, it is apparent the importance to develop innovative agricultural systems and to promote technologies that could optimize the exploitation of water resources; nonetheless, it is crucial to guarantee that the appropriate amount of water is timely and efficiently delivered to the plants. As reported in the related literature, a number of solutions to this problem are being considered [4]. For example, prediction models for irrigation are constantly being developed for facing the needs for short- and long-term water resource management [5]. Moreover, Ines *et al.* [6] combined remote sensing-simulation modeling and genetic algorithm optimization to explore water management options in irrigated agriculture. Indeed, the severity of the subject has motivated constant interest in individuating effective technological and management solutions for water irrigation optimization [7–10].

Another approach to the optimization of water consumption is the use of superabsorbent polymers (SAPs) [11]. SAPs can absorb and retain extremely large amounts of a liquid (water or an organic liquid) relative to their own mass [12].

In agricultural applications, SAP granules are mixed with the soil in given amounts. After watering, the granules absorb the water by swelling, and then release it slowly through a diffusive mechanism, as the soil gets dry. In this way, irrigated water is not lost through drainage or evaporation while being efficiently supplied to the plant roots when needed. Furthermore, SAP granules increase their size upon swelling, thus enhancing soil porosity and providing a better oxygenation to the roots. A further advantage of SAPs in agriculture is that they can be loaded with nutritional substances and phytopharmaceuticals, which are then gradually released to the plants.

Therefore, employing SAP in cultivations would help not only in minimizing water consumption, but also in rationalizing the use of phytopharmaceuticals, especially in the following agricultural systems:

(A) *Protected cultivations*: these cultivations are characterized by great intensity and high specialization of the soil, which ultimately leads to the deterioration of the essential nutrients of the soil, to the accumulation of telluric pathogens and to secondary salinization due to the excessive use of fertilizers and/or brackish water. In this context, not only would the adoption of SAP rationalize the amount of used water, but it would also allow modulating (in time) the supply of nutrients, thus avoiding the accumulation of toxic substances in the soil;

(B) *Soilless cultivations*: in soilless cultivations, plants are grown using mineral nutrient solutions in water, without soil [13]. In open soilless systems, there is a massive waste of water and nutrients, which is responsible for an increase in running costs and in contamination of ground and surface

water [14]. The adoption of the SAP to ration the delivery of nutrients to the plants would improve the overall environmental sustainability of these systems. Also for closed systems (in which water recirculates), the use of SAP may help hindering water retention of the plants;

(C) *Open-field cultivations*: in agriculture, chemically synthesized fertilizers are commonly supplied in larger quantity than actually needed by the plants. Nitrate accumulation in the soil, especially in dry areas, is a typical result of such an excessive use of fertilizers, with negative repercussions on the environment as well as on the product quality, with nitrates being absorbed by the plants and fruit. The presence of nitrates decreases the nutritional quality of the product (especially for leaf vegetables), and increases the risk of developing cancers [15]. The use of certain SAP formulations could allow reducing the amount of fertilizers used and/or limiting nitrate accumulation in the soil.

Pioneering experiments carried out by a Japanese company in the Egyptian desert, in the early 1990s, demonstrated the potential of synthetic SAPs for water management in agricultural applications [16]. Similarly, Woodhouse and Johnson [17] used synthetic SAPs as soil conditioners to aid plant establishment and growth in drought-prone growing media. The most efficient and most widely employed SAPs are based on polyacrylates, *i.e.*, non renewable materials derived from petroleum industry that are reported to degrade at rates less than 10% per year, via delamination, shear-induced chain scission and photosensitive chain scission [18]. Because of their very low degradation rate, acrylic SAPs are regarded as potential pollutants for the soil. Moreover, there are also some concerns regarding the release of toxic molecules during their slow degradation. As a result, in the last decade, the increasing interest in environmental issues has led manufacturers and researchers to focus on the development of alternative, environmentally friendly SAPs. Examples of investigated biopolymers for the synthesis of SAPs include cellulose derivatives [19] and starch [20], which can be degraded by soil microbes [19,21].

In this work, an innovative SAP, obtained through chemical crosslinking of cellulose derivatives, was synthesized, based on the results of previous studies [22–30], and its water retaining capability was assessed with a specific focus on agricultural applications. The goal of this work was to characterize, from an operative point of view, the beneficial effect of the use of the novel SAP in combination with different types of soils, for the cultivation of plants typical of the Mediterranean area. To this purpose, preliminary experiments were carried out to evaluate the absorption capacity of the SAP. Successively, for assessing the efficiency of the SAP after several water absorption/desorption cycles in the soil, the SAP was tested (in different concentrations and at different depths in the soil) for the cultivation of plants inside pots. Finally, the SAP was employed in conditions that mimicked open-field cultivations, in order to analyze its effect on the growth of plants.

2. Materials and Methods

In order to test the suitability of the cellulose-based SAP for use in agricultural applications, the following experiments were carried out:

- (i) Characterization of the absorption capacity of the SAP in both distilled and tap water;
- (ii) Evaluation of the effect of the SAP when placed in different types of soil and at different depths, through experiments performed in plant pots; and

(iii) Evaluation of the effect of the SAP in conditions that resembled open-field cultivations.

2.1. Synthesis of Cellulose-Based SAP

For the synthesis of the SAP, cellulose derivatives meeting food and pharmaceutical standards were used. In particular, Carboxymethylcellulose sodium salt (CMCNa) and hydroxyethylcellulose (HEC) were the precursors adopted for the hydrogel synthesis. The detailed description of the synthesis process of the used SAP, which is based on the results obtained in previous works [23–30], can be found in [22]. The final powder size distribution of the SAP was in the range 0.1–1 mm. Cytotoxicity tests performed in previous studies showed that the SAP does not exhibit any toxicity [31].

2.2. Preliminary Characterization of the Absorption Capacity of the SAP

For the intended application, it is important to study and characterize the absorption characteristics of the material in water and in saline solutions with different ionic strengths, thus simulating the actual conditions in which the SAP is used (namely, in contact with nutrients, fertilizers, and soil). A preliminary experiment was conducted to assess the absorption capacity per gram of the used SAP. To this purpose, two samples of SAP weighing approximately 1 g were added with 100 g of water (W_{add}) each. After 24 h, during which the SAP had reached water saturation, each sample was passed through a membrane filter. This allowed to separate the excess water (*i.e.*, the water that had not been absorbed), whose weight ($W_{not-abs}$) was measured through a precision electronic balance. The weight of the water absorbed by the SAP (W_{abs}) was then evaluated as $W_{add} - W_{not-abs}$.

A second experiment was performed to evaluate the effect of the environmental electrical conductivity on the swelling properties of the SAP. Indeed, due to the polyelectrolyte nature of CMCNa, the absorption capacity of the SAP strongly depends on the dissolved salts that are present in the solvent: the higher the amount of salts, the lower the absorption capacity. This aspect is crucial for agriculture-related applications. Generally, the electrical conductivity of dry soils is almost negligible. However, the value of electrical conductivity increases with the water content in the soil, due to the progressive dissolution of salts. Furthermore, it is necessary to take into account the electrical conductivity of water itself and of fertilizers that are typically used in agriculture [32]. In this work, comparative absorption tests were carried out by mixing 4 g of SAP with both distilled and tap water. The electrical conductivity of tap water was 1000 $\mu\text{S}/\text{cm}$, as measured through a conductivity meter (model HI 9811-5). Every 24 h for seven days, the excess water was removed from the samples and weighed, as described above. This allowed to assess not only the initial amount of water absorbed by each sample, but also the decrease of water-saturation (s_{SAP}) in time.

2.3. Selection of Soils and Types of Plants

To characterize the behavior of the SAP in the agriculture setting, two different types of soil were selected: red soil (whose color is due to the presence of iron compounds), and white soil (which contains a high amount of clay). The chemico-physical characteristics of the used soils are summarized in Table 1. Moreover, two types of plants were chosen for cultivation: chicory (*Cicoria otrantina*) and tomatoes

(*Pomodoro di Morciano di Leuca*). Such plants are typical of the Mediterranean areas and grow during the summer season, which makes them particularly suitable for testing the effectiveness of the SAP in critical conditions.

Table 1. Characteristics of the two types of soil employed in the experiments.

Parameter	White soil	Red soil
pH	8.21	8.35
Electrical conductivity	154.3 $\mu\text{S/cm}$	146.9 $\mu\text{S/cm}$
Organic content	17,613.75 mg/kg (1.76%)	24,593.88 mg/kg (2.46%)
N (total)	0.784 g/kg	1.008 g/kg
Na	4.11 meq/100 g	2.77 meq/100 g
Ca	100.798 meq/100 g	133.03 meq/100 g
Mg	12.86 meq/100 g	16.45 meq/100 g
K	2.65 meq/100g	3.78 meq/100 g
Assimilable P ₂ O ₅	203.99 meq/100 g	195.72 meq/100 g
Limestone (total)	4%	8%

2.4. Experimental Setup for the Evaluation of the Effect of the SAP in the Soil

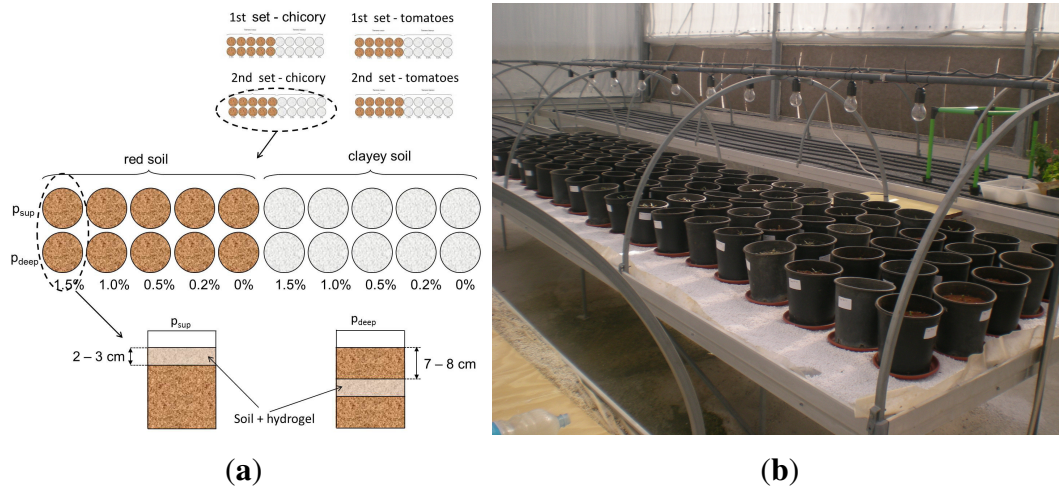
A total of eighty pots filled with SAP-amended soil were prepared. Figure 1a,b show a schematization and a picture of the experimental setup, respectively. As can be seen from Figure 1a, four sets of samples were prepared: two sets were planted with chicory and two sets with tomatoes. Each set consisted of 20 pots (top schematization of Figure 1b). Each 20-pot set consisted of 10 pots filled with red soil and 10 pots filled with red soil (central schematization of Figure 1a). In turn, each subset of 10 samples was subdivided in five pairs: a pre-established weight percentage of SAP (w_{SAP}) was added to each pair of samples, namely 0%, 0.2%, 0.5%, 1.0% and 1.5% of SAP. For each of these pair of samples, in one pot the SAP was mixed with the surface/near-surface soil (p_{sup}); whereas in the other pot the SAP was mixed deeper within the soil (p_{deep}), as depicted in the bottom schematization of Figure 1a.

The effect of SAP on the cultivations was then evaluated according to the following procedure. Each sample was weighed before (*i.e.*, in the dry state) and after watering with one liter of water. Starting from the latter condition, the percentage water content (θ_s) of each sample was checked regularly gravimetrically, and monitored over 78 days. During this period, further addition of water was performed only when either of the following conditions occurred:

- (i) the value of θ_s in the sample without SAP (*i.e.*, the reference sample, $w_{\text{SAP}} = 0$) had decreased to 10%; or
- (ii) for all the samples, the evaporated water equaled the percentage of water evaporated from the reference sample.

This experiment, which was carried out inside a greenhouse, allowed assessing whether the presence of the cellulose-based SAP was actually effective in preventing (or at least hindering) natural evaporation phenomena of the supplied water.

Figure 1. Schematization of the 80 pots that were prepared. The figure also indicates the different weight percentage of Superabsorbent polymer (SAP) and the portion of soil that was mixed with the SAP (a); Picture of the plant pots as prepared for the experiment (b).

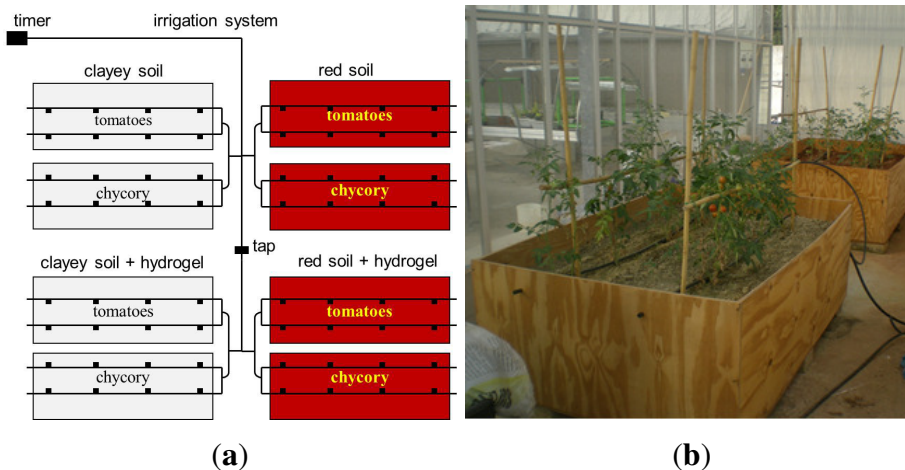


2.5. Experimental Setup for Mimicking Open-Field Cultivations

After the plants had been grown in the seedbed, they were transplanted in large boxes, thus somewhat resembling an open-field cultivation. Also this experiment was carried out inside a greenhouse, from late spring to August; hence, the environmental temperature was particularly high.

The experimental setup consisted of eight wooden boxes (with dimensions 1.80 m × 0.80 m × 0.60 m) filled with soil. Four boxes were cultivated with tomatoes and four boxes with chicory. For each type of plant, two boxes were filled with red soil and two with white soil. In turn, for each type of plant and for each type of soil, one box contained plain soil, whereas the other box contained soil with SAP. In each box, eight plants were planted. Figure 2a shows a schematization of the disposition of the wooden boxes. Figure 2b shows a picture of the experimental setup.

Figure 2. Schematization of the disposition of the boxes. A tap was used to reduce the amount of water delivered to the “soil plus SAP” boxes (a); Picture of the experimental setup (b).



It is important to point out that the SAP was added only on the surface-layer of the soil, in correspondence of each plant (5 g of SAP for each plant).

Each box was equipped with a drip irrigation system. In order to test the effectiveness of the SAP, the plants in the “soil-plus-SAP” boxes were watered with half the amount of water that was supplied to the plants without SAP. In fact, every 12 h, each dripper released approximately 0.33 L of water to the plants in the “soil-plus-SAP” boxes and 0.66 L to the plants in the boxes without SAP. The growth of the plants was visually observed over a period of three months.

3. Experimental Results

3.1. Absorption Capacity of the SAP

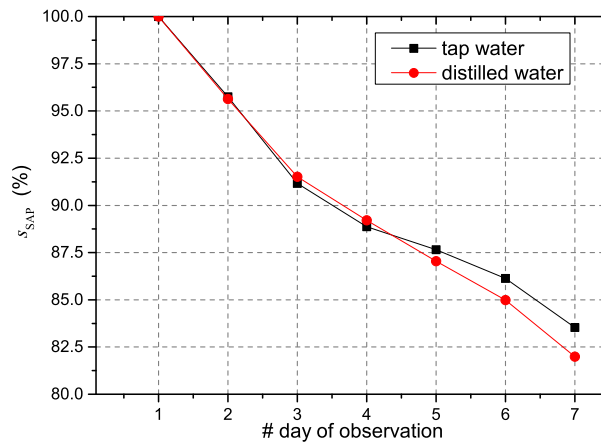
The swelling tests in distilled water showed that the cellulose-based SAP can absorb water up to 74 times its own weight (Table 2). This value, which is comparable with those reported for other biopolymer- [19] and acrylic-based [33] SAPs, is clearly appealing for the intended application.

Table 2. Summarized results of the experiment for assessing the absorption capacity of the SAP.

Sample	SAP (g)	$W_{add}(g)$	$W_{not-abs}$ (g)	W_{abs} (g)
#1	1.01	100.04	25.40	74.64
#2	1.00	100.06	25.30	74.76

The second swelling experiment performed in this study focused on the effect of environmental electrical conductivity on the absorption capacity of the SAP as well as on the subsequent water loss kinetics from the hydrogel. In particular, a comparison was made between a sample of SAP saturated with tap water and another sample saturated with distilled water. Starting from the water-saturated condition, the desaturation of the SAP was then monitored for seven days by weighing the amount of water that was released daily. While the absorption of the SAP in distilled water was about 74 g water/g SAP, as reported above, the absorption in tap water was about 40 g water/g SAP, which fits in the range of the typical absorption capabilities of SAPs in saline media (30–60 g water/g SAP) [32]. Furthermore, as shown in Figure 3, in the first 4 days the desaturation behavior was similar for the two samples, independently of the type of water used. However, starting from the 5th day of observation, the percentage of water released by the SAP saturated with tap water was lower than that released by the SAP saturated with distilled water. This may be an indicator of the fact that, although the SAP absorbs a lower amount of water when it is in an electrically conductive-solvent (such as tap water), its water retaining capability is higher, likely due to the presence of fixed electrostatic charges on the polymer network which contribute favourably to the overall interaction between the solvent and the SAP. This finding thus further suggests the potential of the SAP for agricultural applications.

Figure 3. Water desaturation of the two samples of SAP mixed with distilled water and with tap water, in the seven-day observation period.



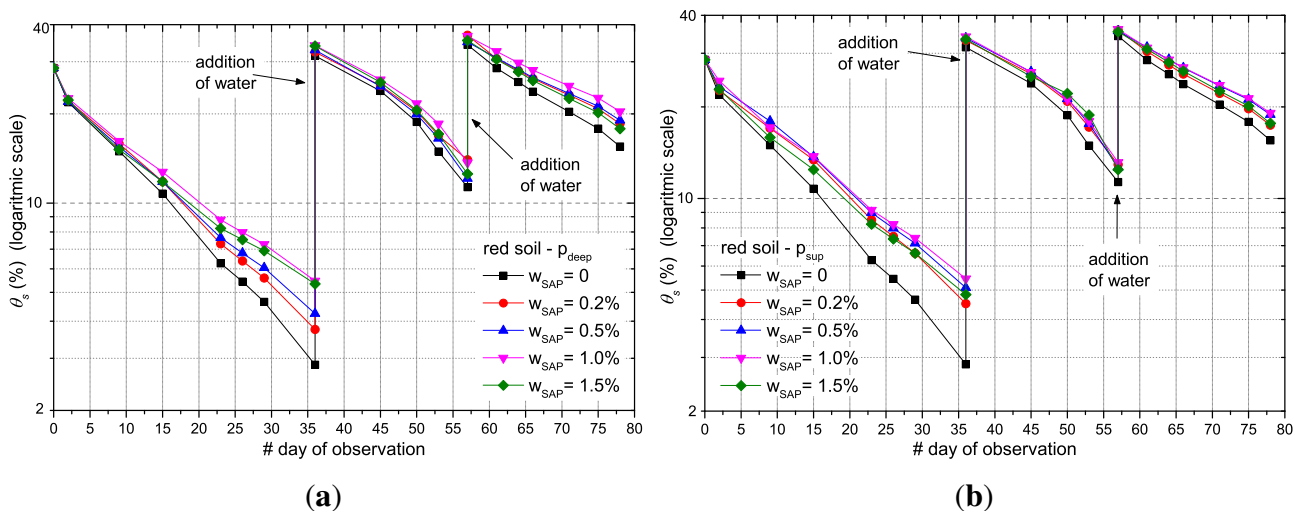
3.2. Evaluation of the Efficiency of the SAP after Several Water Absorption/Desorption Cycles

The percentage gravimetric moisture content θ_s of the soil in the pots was monitored regularly. The addition of water to the pots was necessary only at the following time points:

- (1) on the 36th day, one litre of water was added to both types of soil;
- (2) on the 57th day, 0.8 L of water was added only for the red soil;
- (3) on the 64th day, 0.5 L of water was added only to the clayey soil.

For the two types of soil, and for each concentration of SAP, Figures 4 and 5 show the variation of θ_s during the observation period, for different values of w_{SAP} . In the figures, the abrupt changes of θ_s correspond to the aforementioned additions of water.

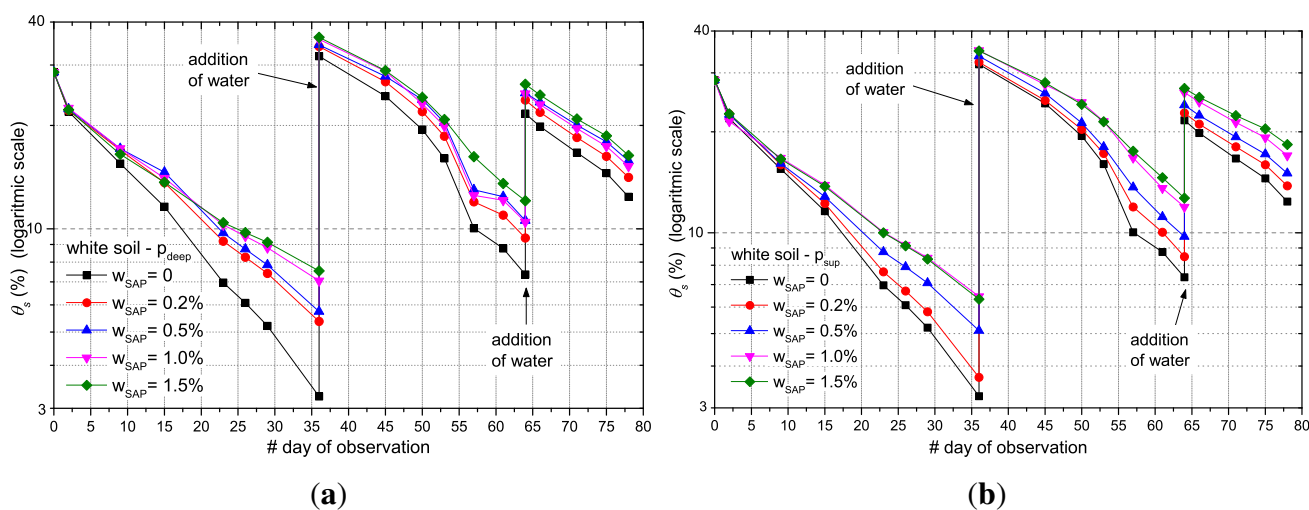
Figure 4. Variation of the moisture content of the red soil during the 78-day observation time: for p_{deep} (a) and p_{sup} (b) samples, with different percentages of SAP.



From Figures 4 and 5, it can be seen that value of θ_s in the pots free of SAP is always consistently lower than in the pots containing SAP. This confirmed that the presence of the SAP had a beneficial

effect in hindering water evaporation phenomena and in providing a higher quantity of water over a longer period of time. Furthermore, at any given day of observation, it can be seen that a higher amount of SAP (hence, a higher value of w_{SAP}) generally led to a higher amount of moisture content in the soil. Indeed, for red soil, this effect was not evident for the case of $w_{SAP} = 1.5\%$; in fact, in this case, the corresponding values of θ_s were slightly lower than the values corresponding to $w_{SAP} = 1.0\%$. This was probably attributable to a “self-saturation” effect of the SAP, which ended up retaining water within itself (rather than releasing it).

Figure 5. Variation of the moisture content of the white soil during the 78-day observation time: for p_{deep} (a) and p_{sup} (b) samples, with different percentages of SAP.

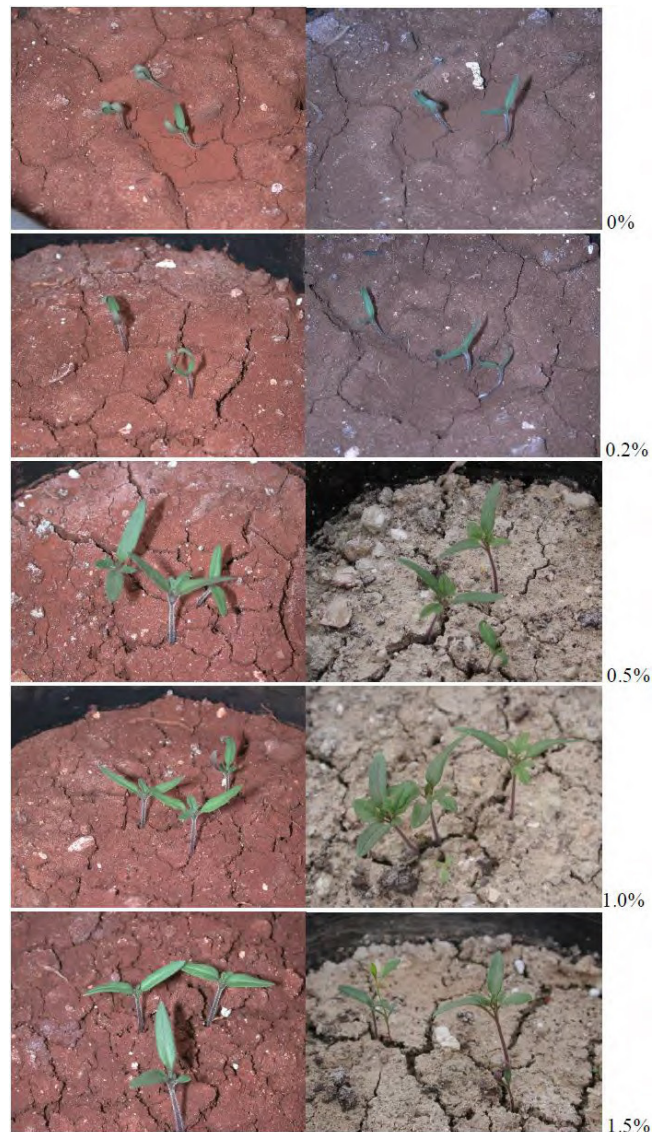


Another important conclusion that can be drawn from these results is that the efficiency of the SAP decreased with time. In fact, for all the w_{SAP} values, the period of time between the first addition of water and the second addition of water was 15 day and 8 day shorter, for red soil and clayey soil, respectively. This is a direct result of the fact that after the first addition of water (at the 36th day), the SAP had probably started to degrade, thus losing its water retaining efficiency.

Overall, the obtained results demonstrated that the proposed SAP could guarantee a good performance for approximately 80 days. In practical applications, the use of fertilizers (containing nitrogen, phosphor, potassium and calcium) causes an increase of ions, and thus an overall decrease of the retaining capacity of the SAP. This would likely induce the reduction of the expected effectiveness time of the SAP, to values of about 60 days. However, this would still represent an important achievement for the intended application.

Figure 6 shows a visual comparison of the growth of tomatoes plants for different concentrations of SAP. For both types of soil, the plants with the higher SAP percentages looked healthier than those grown in the soil without the amendment of the SAP or with lower SAP percentages. In particular, for the lowest SAP concentration tested ($w_{SAP} = 0.2\%$), the leaves of the plants appeared small, likely because the SAP quantity was not sufficient to reduce water evaporation and to preserve an appropriate amount of water in the soil. Similarly, the leaves of the plants grown in the absence of the SAP were particularly small and wrinkled.

Figure 6. Visual comparison of the growth of tomatoes plants for the samples with different concentration of SAP, for red soil (**left side**) and white soil (**right side**). In these plants, the SAP was mixed with the surface layer of the soil.



3.3. Experimental Results for the Cultivation Mimicking Open-Field Conditions

The growth of the plants transplanted in large wooden boxes (as described in Section 2.5) was observed regularly for approximately three months, and in this period, no phytosanitary treatment was carried out. Overall, in this observation period, 120 L of water were supplied to each plant in the plain soil, and 60 L to each plant in the soil with SAP.

Figures 7 and 8 show how the chicory and the tomato plants had grown in the considered conditions. In spite of the strong reduction of the supplied water, the plants in the boxes amended with SAP grew similarly to the plants in the boxes without SAP (for example, the tomato plants gave approximately the same amount of fruit). Only one row of chicory grown in the SAP-amended red soil did not survive; however, it is safe to assume that this was not related to the SAP (also considering that the other row of chicory in the same box grew regularly).

Figure 7. Comparison of the growth of chicory plants in the different types of soil, with and without the addition of SAP.

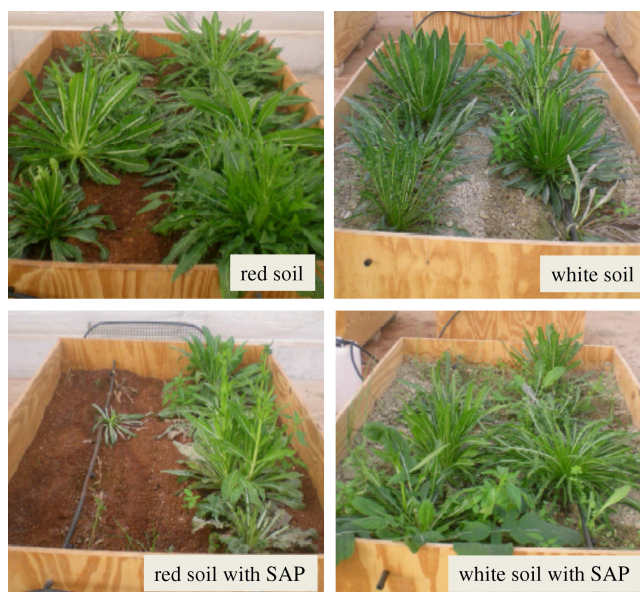


Figure 8. Comparison of the growth of tomato plants in the different types of soil, with and without the addition of SAP.



The obtained results thus demonstrated the effectiveness of the SAP in view of application in open-field cultivations. In this regard, it is worth emphasizing that the considered types of plants need substantial amount of water, and that a 50%-reduction of water (to the plants with SAP) was an

intentionally critical condition to demonstrate the beneficial effect of the SAP. This is particularly true considering that this experiment was carried out in summer, inside a greenhouse, in harsh environmental conditions (the temperature often exceeded 45 °C).

4. Conclusions

The employment of an innovative class of cellulose-based SAPs for optimizing water consumption in agriculture was assessed. The used SAP exhibited absorption capacities suitable for the envisaged application. The water-retaining properties of the SAP were studied through several experiments that allowed evaluate the beneficial effect on the optimization of irrigation. Indeed, the obtained results showed that the addition of SAP to the soil delays water evaporation, thus making water available to plants over a longer period of time. The experiments performed in conditions simulating open-field cultivations showed that, in spite of a significantly sharp reduction of supplied water (50%) for SAP-amended soils, the plants grew regularly. The proposed SAP thus show promise for rationalizing water consumption in agricultural irrigation, while possibly overcoming the environmentally critical limitations of acrylate-based, state-of-the-art SAPs.

Author Contributions

Giuseppe Cannazza and Egidio De Benedetto designed the experiment and conducted the experimental work. Christian Demitri and Marta Madaghiele designed and realized the innovative SAP. The paper was prepared under the direction, review and guidance of Andrea Cataldo and Alessandro Sannino, for the experimental part and for the design/realization of the SAP, respectively.

Conflicts of Interest

The authors declare no conflict of interest.

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