soil systems

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Water Retention Characteristics of Superabsorbent Polymers (SAPs) Used as Soil Amendments

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Abstract: Superabsorbent polymers (SAPs) are used as a soil amendment for retaining water, but suitable methods for the application of SAPs have not yet been developed. Here, we characterized a variety of soil–SAP mixtures prepared using four different types of SAP in terms of their water absorption and release characteristics. The teabag method was applied to characterize the soil–SAP mixtures, except for measurements of the matric potential. The results showed that the variations in water absorbency among the four SAPs in isolation became insignificant when they were mixed with sandy soils. The rates of water released from the soil–SAP mixtures under heated conditions were mitigated with decreasing water content, which prolonged the time until desiccation of the mixtures. The water absorbency of the SAPs significantly decreased in salt solutions (KCl and $CaCl_2$), but their absorbency mostly recovered following immersion in tap water. The soil–dry SAP mixtures retained a larger amount of water than the soil–gel SAP mixtures. Swollen SAPs predominantly retained water in the range of −0.98 to −3.92 kPa, suggesting that SAP induces a transition from gravitational water to readily plant-available water by swelling itself. SAPs barely increased the amount of plant-available water in a potential range of −3.92 to −98.1 kPa, but significantly increased the soil water at <−98.1 kPa. The soil water content increased with an increasing SAP application rate, whereas the proportion of plant-available water declined. Our findings indicated that the performance of SAPs depends on the pore space and a saline environment in the soil and that low SAP application rates are suitable for maximizing the water available to plants in sandy soils.

Keywords: evaporation; hydrogel; soil matric potential; saline soils; sandy soils

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1. Introduction

Increases in global population, climate change, and desertification have accelerated the problems of declining crop productivity, devastation of agricultural lands, deforestation, and expansion of poverty. In dry and semidry regions, deficits in plant-available water are the most significant factor inhibiting agricultural activities, biomass production, and afforestation; thus, soil water management is the key to success [1]. Sandy soils, such as Psamments and Arenosols, have poor water-holding capacity and are widely distributed in tropical regions [2,3]. Although various soil conditioners such as organic manure and biochar can be used to improve the water retention characteristics of sandy soils [4–8], superabsorbent polymers (SAPs) are sometimes used as a relatively new alternative [9–11]. Because of its high water-absorbing capacity, SAP results in improvements in germination rate, seedling survival rate, production of crops, fruit, and vegetables, and forestry [12–14]. However, negative results of SAP application have also been reported in field trials [15–18]. Complex interactions among soil conditions, plant species, and SAP products make it difficult to understand the effects of SAP on plant growth. As a consequence of recently changing climate conditions, the development of suitable SAP application methods is now required to stabilize agriculture and forestry [19].

In spite of the promising results of biodegradable and natural organic polymers in environmentally friendly agriculture systems [20,21], most commercial SAPs are still synthetic 3D cross-linked polymers composed of synthetic chemicals such as polyacrylic acid, polyacrylamide, and vinyl alcohol. A variety of SAPs, i.e., those with different polymer materials, cross-linking densities, counterions, and grain sizes, are available in the market, each showing different characteristics in terms of water-absorbing capacity and durability [22,23]. However, it has been reported that the characteristics of SAP alone do not always translate to effective soil amendment for improving water retention [24,25]. Several studies have reported suitable methods for applying SAPs to soils, including, for example, altering the concentration of SAPs, the use of dry SAPs or hydrated gel SAPs, altering the depth of application of the SAP, and altering the watering procedure [9,15,23,25,26]; however, their application to farmland and commercial plantations has rarely been reported. Thus, suitable techniques of SAP application for soil amendment still await development, which would also be useful for biodegradable SAPs.

The application of SAPs to soils as a soil amendment or conditioner has a long history, with general agreement on the characteristics of SAPs found in several academic reports and reviews [11,13,27–29]. For example, the effects of SAPs are more prominent in soils with sandy rather than clayey textures [30,31], and salts in the soil and irrigation water diminish the water absorbency of SAP, especially for multivalent cations [32,33], which are related to the physical and chemical swelling mechanisms of the active polymer in the SAP. Thus, the possibility of applying SAPs is of interest in salt-affected lands in tropical dry areas [21,30,34]. Furthermore, because SAPs usually show high levels of water absorbency, the availability of water for herbs and trees is also of interest. The matric potential of water retained in SAPs is often examined to evaluate the plant availability of SAPs' water [29,30,35,36].

The characterization of SAPs using conventional laboratory experiments is challenging and sometimes fails because the volume of the soil and SAP mixtures varies, as the mixtures mainly swell and shrink in an unexpected manner during the experiments [24,37,38]. Thus, quantitative data based on a constant volume—a unit for soil physics—cannot often be obtained by the standard methods using standard equipment. Special apparatus, such as a Richards pressure cell [39] or a chamber with a force sensor [40], can be applied. However, the soil volume with a SAP varies depending on the soil moisture conditions and the soil depth to which the SAP has been applied in the fields [30,41]. Thus, interpretation is needed to apply the experimental data to actual field conditions, even though precise laboratory data have been obtained. It would be meaningful to develop simple and tractable methods for characterizing the performance of SAPs because SAP is a tricky material for accurate experiments. The teabag method has often been used to evaluate the water absorbency of individual SAP products [42–46]. As a simple experimental method, the teabag method may also be applied to assess the characteristics of SAP as a soil amendment, even though the data may be semiquantitative under limited conditions in the bag.

This study aimed to understand the essential characteristics of SAP products as soil amendments for retaining and releasing water. Four types of SAP products and standard sand were used as experimental materials. A series of experiments was conducted in the laboratory using the SAPs in isolation and soil–SAP mixtures. The teabag method was applied to examine the water absorbency of the SAPs and the water released from SAPs by heating. The effects of salt solutions on SAP absorbance were also examined under various conditions. The conventional methods of a sand column and a pressure chamber were used for measuring the matric potential of water in soil–SAP mixtures. Changes in the plant availability of water in soil–SAP mixtures were evaluated according to different SAP application methods and rates. At the end of this paper, suitable SAP application methods in the field are discussed.

2. Materials and Methods

2.1. SAP and Soil

All four types of SAP studied were cross-linked polymers made of acrylic acid polymer or acrylamide and acrylic acid copolymers: GT-1 (SANFRESH GT-1, Sanyo Chemical Industries, Ltd./SPD Global Co. Ltd., Kyoto, Japan), ST (SANFRESH ST-500D, Sanyo Chemical Industries, Ltd./SPD Global Co. Ltd., Japan), PAAK (a prototype product by Sanyo Chemical Industries Ltd., Kyoto, Japan), and AQS (AQUASORB™ 3005 K4, SNF group, Andrézieux-Bouthéon, France). These are white, odorless powders in dry condition. Table 1 shows the characteristics of the SAPs, which differ in terms of their polymer material, counterions, grain size, and density. Dry SAPs were used for experiments unless otherwise stated.

Table 1. Characteristics of the superabsorbent polymers (SAPs) used in the experiments.

SAP	Polymer	Countercation	Bulk Density (Mg m^{-3})	Grain Diameter
SANFRESH GT-1 (GT-1)	Acrylic acid	nondisclosure	0.36	150–500 µm
SANFRESH ST-500D (ST)	Acrylic acid	Na	0.62	300–500 µm
A prototype product (PAAK)	Acrylic acid	K	0.69	300–500 µm
AQUASORB™ 3005 K4 (AQS)	Acrylic acid + acrylamide	K	0.75	1.7–2.8 mm

The soil used was sand no. 5, guaranteed by the Japan Industrial Standards (JIS), which mainly consists of silica sand. The proportions of sand particle sizes in the ranges 1–0.5, 0.5–0.25, and 0.25–0.1 mm were 70.1%, 28.9%, and 1.0%, respectively. True specific gravity was 2.65 Mg m^{-3} and dry bulk density was 1.52 Mg m^{-3}, indicating 57.4% solid phase and 42.6% air phase in the dry bulk density.

The soil–SAP mixtures were prepared by thoroughly mixing specific amounts of SAP and soil. The SAP application rate in the soil was calculated as follows:

$$\text{SAP application rate (\%)} = [\text{dry weight of SAP}]\ (\text{g})/[\text{dry weight of soil}]\ (\text{g}) \times 100 \quad (1)$$

2.2. Immersing Solutions

The SAPs were immersed in the solutions to examine their water absorbency. Pure water, tap water, and salt solutions were used in the experiments. The salt concentrations of the solutions were based on the references for irrigation water quality [47,48]. The concentrations of KCl were 10 and 20 mM, and those of $CaCl_2$ were 5 and 10 mM. Tap water was used in the experiments on the soil–SAP mixtures. Solution pH and electric conductivity (EC) were as follows: pH 5.4 and 0 µS/cm for pure water; pH 6.7 and 188 µS/cm for tap water; pH 5.3 and 2.3 mS/cm for 10 mM KCl; pH 5.4 and 4.6 mS/cm for 20 mM KCl; pH 5.5 and 2 mS/cm for 5 mM $CaCl_2$; and pH 5.5 and 3.8 mS/cm for 10 mM $CaCl_2$.

2.3. Water Absorbency of SAP and Soil–SAP Mixtures

The water absorbency of SAP was determined using the method described in JIS-K7223-1996 [43]—the so-called teabag method. The teabag was made of a nylon 250-mesh sheet 10 cm wide and 20 cm deep. A certain weight of SAP or soil–SAP mixture was placed into the teabag. The bag was hung with a string attached to its upper corner and immersed in a 1 dm^3 solution. The bag was then removed from the solution and suspended until any dripping water ceased to weigh the bag down (approximately 3 min). The measurements were performed every 2–3 h. The water content in the SAP or soil–SAP mixture was calculated using the following equations:

$$\text{SAP water content} = [\text{water absorbed by SAP}]\ (\text{g})/[\text{dry weight of SAP}]\ (\text{g}) \quad (2)$$

$$\text{Soil water content} = [\text{water content of soil-SAP mixture}]\ (\text{g})/ [\text{dry weight of the soil-SAP mixture}]\ (\text{g}) \quad (3)$$

The maximum water-holding capacity of the SAP alone was defined as the water content in the SAP determined at 24 h after immersion in the solution. The maximum water-holding capacity of the soil–SAP mixture was defined in the same manner, i.e., the water content in the soil–SAP mixture at 24 h after immersion.

2.4. Water Release from SAP by Heating

After measuring the absorbency of the SAP or soil–SAP mixture, as described in Section 2.3, the teabag was hung in a heating box (63 cm × 43 cm × 45 cm) at 60 °C and left to dry, assuming the soil surface condition to be that in the tropical region. The bag was weighed every 2 or 3 h to determine the water release rate by evaporation from the bag until the soil–SAP mixture was completely desiccated. The evaporation rate (g g^{-1} h^{-1}) of the soil–SAP mixture was calculated as follows:

$$\text{Evaporation rate of soil-SAP mixture} = (\text{Wtb}(\text{t1}) - \text{Wtb}(\text{t2}))/\text{Ws}/(\text{t1} - \text{t2}) \quad (4)$$

where Wtb(x) is the weight (g) of soil–SAP mixture at a time x, t is time (hour), and Ws is dry weight of soil (g).

2.5. Soil Water Retention Curves and Plant-Available Water

The soil water potentials of the soil–SAP mixtures and the soil alone were determined using the sand column method (DIK-3521, Daiki Rika Kogyo Co., Ltd., Saitama, Japan) for a potential range of −0.98 to −3.92 kPa, equivalent to a soil pF range of pF 1.0–1.6 (gravitational water), followed by the pressure chamber method (DIK-3404 chamber, and DIK-9222 pressure controller, Daiki Rika Kogyo Co., Ltd., Japan) for a range of −3.92 to −98.1 kPa, equivalent to a soil pF range of pF 1.6–3.0 (from field capacity to readily plant-available water). The rest of the water consisted of medium to barely plant-available water (−98.1 kPa to −1500 kPa) and unavailable water (<−1500 kPa). Because the soil–SAP mixtures swelled when they absorbed water, the volume of the soil sample examined was variable, depending on the SAP application rate and the water content. Thus, the soil–SAP mixtures were placed in a 100 cm^3 soil sampling core (5 cm in diameter and 5 cm in depth, covered with a paper filter at the bottom) to a depth of 3 cm at most. The initial water conditions for the retention measurements were prepared by placing the core on the surface of the sand column, which was adjusted to a pressure of −0.98 kPa, for 24 h. Water saturation could not be determined because the volume of soil–SAP mixtures was not constant. Despite swelling over the top edge of the core, none of the soil–SAP mixtures fell off during the experiment owing to careful operation. Because the volume of the soil–SAP mixtures changed during measurement, the soil water content is expressed on the basis of dry soil weight.

2.6. Experiments for Characterizing SAP Alone and Soil–SAP Mixtures

The experimental conditions and variables examined in each experiment were summarized in Table 2.

Table 2. Dependent variables, independent variables, and conditions of the experiments.

Experiment	Dependent Variable	Independent Variable	Material	Solution	Method
1	SAP water content	SAP types, solution types, time	SAP	Pure water, 10 mM KCl, 5 mM $CaCl_2$	Teabag
2	SAP water content	SAP type, types and concentrations of salt solution	SAP	10 mM KCl, 20 mM KCl, 5 mM $CaCl_2$, 10 mM $CaCl_2$, tap water	Teabag
3	Soil water content	SAP types, salt types, time	Soil–SAP mixture	Tap water, 10 mM KCl, 5 mM $CaCl_2$	Teabag
4	Soil water content	SAP application rates	Soil–SAP (GT-1) mixture	Tap water	Teabag
5	Water release rate, time to desiccation	Soil water content	Soil–SAP mixture		Teabag
6	Soil water content	SAP types, water potential	Soil–SAP mixture	Tap water	Sand column, pressure chamber
7	Soil water content	SAP types, water contents of SAP gels, water potential	Soil–gel SAP mixture	Tap water	Sand column, pressure chamber
8	Soil water content	SAP application rates, water potential	Soil–SAP (GT-1) mixture	Tap water	Sand column, pressure chamber

2.6.1. Experiment 1: Comparisons of the SAPs' Water Absorbency in Pure Water and Salt Solutions

To compare the water absorbency of the four SAPs in pure water, 0.2 g of each SAP was put into a teabag and its water absorbency and maximum water-holding capacity were measured by the method described in Section 2.3. For the salt solutions, 1 g of each SAP was placed into each of two teabags following the JIS method [43], and the bags were immersed in a solution of each 10 mM KCl or 5 mM $CaCl_2$. The teabags were weighed 3 and 24 h after immersion (Table 2).

2.6.2. Experiment 2: Effects of Salt Types and Concentrations on the SAPs' Water Absorbency and Recovery of Absorbency Using Tap Water

To examine the effects of the type and concentration of salt, four teabags, each containing 1 g of SAP, were immersed in one of the four salt solutions: 10 mM KCl, 20 mM KCl, 5 mM $CaCl_2$, and 10 mM $CaCl_2$. The maximum water-holding capacity of each SAP was determined by the method described in Section 2.3. After determination of the maximum water-holding capacity, the teabags were hung in the drying box for desiccation. The bags were measured repeatedly until their weights were constant. Then, the dried teabags were immersed in tap water, and their water absorbency was again measured by the method described in Section 2.3 (Table 2).

2.6.3. Experiment 3: Comparison of Water Absorbency of Soil–SAP Mixtures in Tap Water and Salt Solutions

Four teabags were prepared by adding 0.2 g of SAP to 100 g soil (one type of SAP per bag), followed by thorough mixing. Each mixture was then placed in a teabag to determine its maximum water-holding capacity using the method described above. The bags were immersed in tap water and in a solution of either 10 mM KCl or 5 mM $CaCl_2$. The maximum water-holding capacity of the soil was determined, and the SAP's water content was calculated. Comparisons were made among all four SAP types (Table 2).

2.6.4. Experiment 4: SAP Application Rate and Water-Holding Capacity of Soil–SAP Mixtures

As a representative SAP product, GT-1 was selected and added to the soil at rates of 0, 0.1%, 0.2%, 0.4%, and 0.8%, followed by thorough mixing. The maximum water-holding capacity of the soil–SAP mixture in tap water was measured using the method described in Section 2.3 (Table 2).

2.6.5. Experiment 5: Comparison of the Rate of Water Release from Soil–SAP Mixtures under Heating

The teabags used in Experiment 3 and 4 were placed in the drying box to measure the evaporation rates following the method described in Section 2.4 (Table 2).

2.6.6. Experiment 6: Soil Water Retention Curves of Soil–Dry SAP Mixtures

Dry SAP grains were mixed with soil at a 0.2% application rate. The initial water-holding capacity at a soil water potential of −0.98 kPa and soil water retention curves were determined using the method described in Section 2.5. All four SAP types were compared (Table 2).

2.6.7. Experiment 7: Soil Water Retention Curves of Soil–Gel SAP Mixtures

Hydrated gel SAPs were prepared in 3 water/SAP ratios, i.e., the pure water/SAP ratios were 25, 50, and 100. The SAP application rate was constant: 0.4% for each water/SAP ratio. The initial water-holding capacities at a water potential of −0.98 kPa and the soil water retention curves were determined following the method described in Section 2.5. All four SAP types were examined. Measurements were performed in a single operation (Table 2).

2.6.8. Experiment 8: SAP Application Rate and Soil Water Retention Curves

As a representative SAP, GT-1 was used to prepare soil–SAP mixtures by increasing the application rates of SAP to the soil by 0, 0.1%, 0.2%, 0.4%, and 0.8%. Soil water retention curves were determined using the method described in Section 2.5. The operation was performed in duplicate, and average data were used (Table 2).

2.7. Statistical Analyses

Measurements were performed in triplicate, except in Experiments 7 and 8. The differences between the means of the treatment in Experiments 1 and 3 were tested using repeated-measures of analysis of variance (ANOVA). The effects of the SAP and salt types on the maximum water-holding capacity were evaluated by two-way ANOVA (Experiment 2). The effects of the SAP types and SAP (GT-1) concentrations on the initial water release rate and the time to desiccation (Experiment 5), and the effects of the SAP types on the amount of water in soil–SAP mixtures (Experiment 6) were compared using one-way ANOVA. The means were separated using the Tukey–Kramer HSD. The level of significance was set to $p < 0.05$ in these statistical analyses. Regression analyses were conducted to determine the relationship between the water content in the soil–SAP mixtures and the SAP application rate (Experiments 4 and 8), and the time to soil desiccation (Experiment 5). Statistical analyses were conducted using JMP 9 software (JMP Japan, Tokyo, Japan).

3. Results

3.1. Water Absorption and Release by SAP Alone

3.1.1. Experiment 1

SAP alone swelled in response to rapid water absorption. Three hours after immersion in pure water, the SAPs' water content reached 65–105% of the maximum water-holding capacity (24 h after immersion) (Figure 1). The maximum water-holding capacities were significantly different among the four SAPs: the water contents of the SAPs were 495 g g^{-1} for GT-1, 441 g g^{-1} for ST, 361 g g^{-1} for PAAK, and 296 g g^{-1} for AQS (Table A1). Immersion in the salt solutions led to a substantial decrease in the maximum water absorption capacity for each SAP type, ranging from 136 to 182 g g^{-1} in 10 mM KCl and from 66 to 114 g g^{-1} in 5 mM $CaCl_2$, with no significant differences.

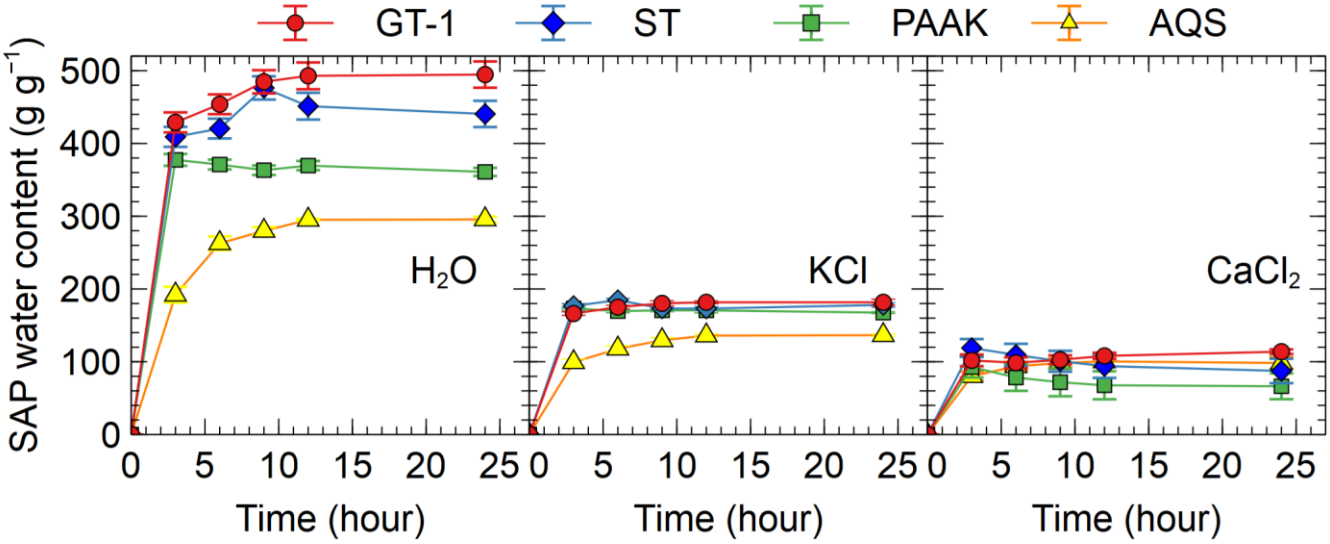


Figure 1. Water absorbency of the SAPs in pure water (**left**), 10 mM KCl solution (**center**), and 5 mM $CaCl_2$ (**right**). Each point shows the mean and standard error (n = 3).

Of the four SAP types, AQS has the largest grain size and showed the slowest water absorption rate and the lowest water-holding capacity. The water contents in ST and PAAK decreased 24 h after immersion in the $CaCl_2$ solution, compared with those measured at 3 h.

3.1.2. Experiment 2

The water contents in SAPs immersed in 10 and 20 mM KCl ranged from 75.6 to 105.6 g g^{-1} and from 56.2 to 85.6 g g^{-1}, respectively (Table 3). The ranges for the SAPs immersed in 5 and 10 mM $CaCl_2$ were 24.6–45.7 g g^{-1} and 5.6–25.0 g g^{-1}, respectively. Absorbency substantially declined in the salt solutions. Stronger ion concentrations decreased the SAPs' absorbency, and the declines were significantly larger with Ca ions than with K ions. However, the effects of salt were not serious for AQS.

Table 3. Maximum water-holding capacity (MWHC) of four SAP types immersed in the salt solutions (10 and 20 mM KCl solutions and 5 and 10 mM $CaCl_2$) and recovery of MWHC through immersion in tap water.

SAP	Salt Solution	MWHC in Salt Solution (g g^{-1})	MWHC in Tap Water (g g^{-1})
GT-1	10 mM KCl	105.0 (1.8) ab	187.2 (4.5) ab
ST		105.6 (1.3) a	177.3 (1.4) ab
PAAK		99.7 (1.2) b	174.6 (3.1) b
AQS		75.6 (0.8) c	137.1 (3.3) c
GT-1	20 mM KCl	82.7 (0.8) a	170.8 (4.4) ab
ST		85.6 (1.2) a	163.3 (1.7) b
PAAK		78.1 (1.2) b	157.2 (1.8) b
AQS		56.2 (0.8) c	121.2 (1.3) c
GT-1	5 mM $CaCl_2$	32.3 (1.6) b	129.0 (5.0) a
ST		26.6 (3.9) b	73.3 (0.4) b
PAAK		24.6 (4.0) b	62.9 (0.3) b
AQS		45.7 (0.8) a	127.2 (0.7) a
GT-1	10 mM $CaCl_2$	7.2 (1.0) b	8.4 (1.0) b
ST		6.5 (1.1) b	4.5 (0.3) c
PAAK		5.6 (1.0) b	3.8 (0.1) c
AQS		25.0 (2.7) a	81.6 (1.3) a

Each value is the mean and the standard error is given in parentheses (n = 3). Means followed by the same letter within a column are not significantly different at $p = 0.05$.

After that, water was released from the teabags by evaporation in the heating box at 60 °C. When the teabags containing dried SAPs were re-immersed in tap water, the SAPs absorbed water again. The maximum water contents of the SAPs in tap water were larger by 1.8- to 4.0-fold compared with those in the salt solutions, and the recovery of absorbency was relatively large for GT-1 and AQS (Table 3). However, with the exception of AQS, the SAPs did not recover their absorbency in tap water when they were first immersed in 10 mM $CaCl_2$.

3.2. Water Absorption and Release of Soil–SAP Mixtures

3.2.1. Experiment 3

The maximum water-holding capacity of soil without SAP was 0.299 g g^{-1} with the teabag method (Table 4). The soil–0.2% SAP mixtures significantly increased their maximum water-holding capacity, ranging from 0.593 to 0.671 g g^{-1}. Compared with SAP alone (Experiment 1), the range of the SAPs' water content was narrow, from 293 to 330 g g^{-1}, and the means of each SAP did not vary significantly. Similarly to Experiment 2, the soil–SAP mixtures quickly absorbed water but later slightly desorbed water: the soil water content at 24 h was 89% of that at 3 h on average. AQS showed a significantly slower water absorption rate, and its maximum water-holding capacity tended to be lower, but not significantly so, compared with other SAP types.

Table 4. Comparison among the SAPs regarding soil water content and the SAPs' water contents in the soil–SAP (0.2%) mixtures at 24 h after immersion in tap water, time to desiccation by heating, and the average evaporation rate in the initial 4 h.

SAP	Soil Water Content ($g\ g^{-1}$)		SAP Water Content ($g\ g^{-1}$)	Time to Desiccation (h)	Evaporation Rate ($g\ g^{-1}\ h^{-1}$)
	3 h	24 h	24 h		
GT-1	0.751 (0.018) a	0.665 (0.067) a	325 (19)	28.7 (0.7) a	0.0418 (0.0043)
ST	0.755 (0.012) a	0.671 (0.051) a	330 (15)	30.0 (1.2) a	0.0378 (0.0060)
PAAK	0.740 (0.012) a	0.650 (0.041) a	322 (12)	26.7 (1.8) a	0.0475 (0.0078)
AQS	0.566 (0.017) b	0.593 (0.018) a	293 (5)	28.0 (1.2) a	0.0503 (0.0044)
no SAP	0.308 (0.012) c	0.299 (0.006) b	No data	11.3 (2.2) b	0.0388 (0.0106)

Each value is the mean and the standard error is given in parentheses (n = 3). Within a column, means followed by the same letter do not differ significantly at $p = 0.05$.

Following immersion in salt solutions of 10 mM KCl and 5 mM $CaCl_2$, the maximum water-holding capacities were lower than those in pure water: the ranges of all four SAP types were from 0.467 to 0.562 $g\ g^{-1}$ for KCl and from 0.307 to 0.345 $g\ g^{-1}$ for $CaCl_2$ (Table 5). The SAPs' water contents at 24 h after immersion ranged from 238 to 282 $g\ g^{-1}$ for KCl, and from 151 to 172 $g\ g^{-1}$ for $CaCl_2$. Especially for the $CaCl_2$ solution, the SAPs' absorbency reduced greatly, and no significant differences in water content were found among the SAP types.

Table 5. Comparison among the SAPs regarding soil water content and the SAPs' water content in soil–SAP (0.2%) mixtures at 24 h after immersion in KCl and $CaCl_2$ solutions.

Salt Solution	SAP	Soil Water Content ($g\ g^{-1}$)	SAP Water Content ($g\ g^{-1}$)
10 mM-KCl	GT-1	0.549 (0.013) a	274 (8) a
	ST	0.540 (0.013) a	276 (7) a
	PAAK	0.562 (0.016) a	282 (8) a
	AQS	0.467 (0.016) b	238 (8) b
5 mM-$CaCl_2$	GT-1	0.307 (0.012)	151 (6)
	ST	0.320 (0.017)	157 (9)
	PAAK	0.322 (0.021)	158 (10)
	AQS	0.345 (0.015)	172 (8)

Each value is the mean and the standard error is given in parentheses (n = 3). Within a column, means followed by the same letter do not differ significantly at $p = 0.05$.

3.2.2. Experiment 4

As the rate of SAP application to the soil increased, soil water content also increased. Linear regression analysis showed that the water content in the soil–SAP mixtures increased linearly, such that the addition of 0.1% SAP resulted in 0.18 $g\ g^{-1}$ water content (Figure 2). In contrast, for GT-1, the SAP water content gradually decreased with increasing SAP application rate from 453 $g\ g^{-1}$ in 0.1% SAP to 216 $g\ g^{-1}$ in 0.8% SAP.

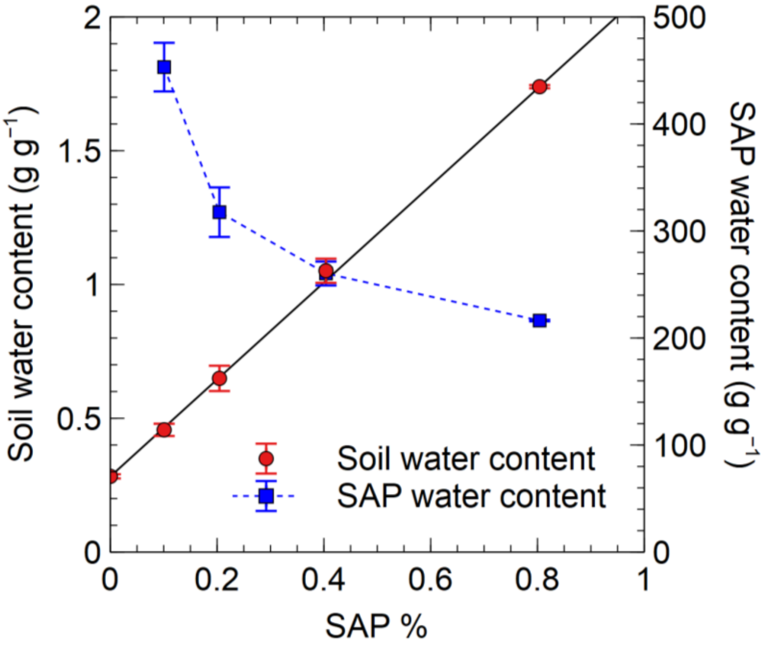
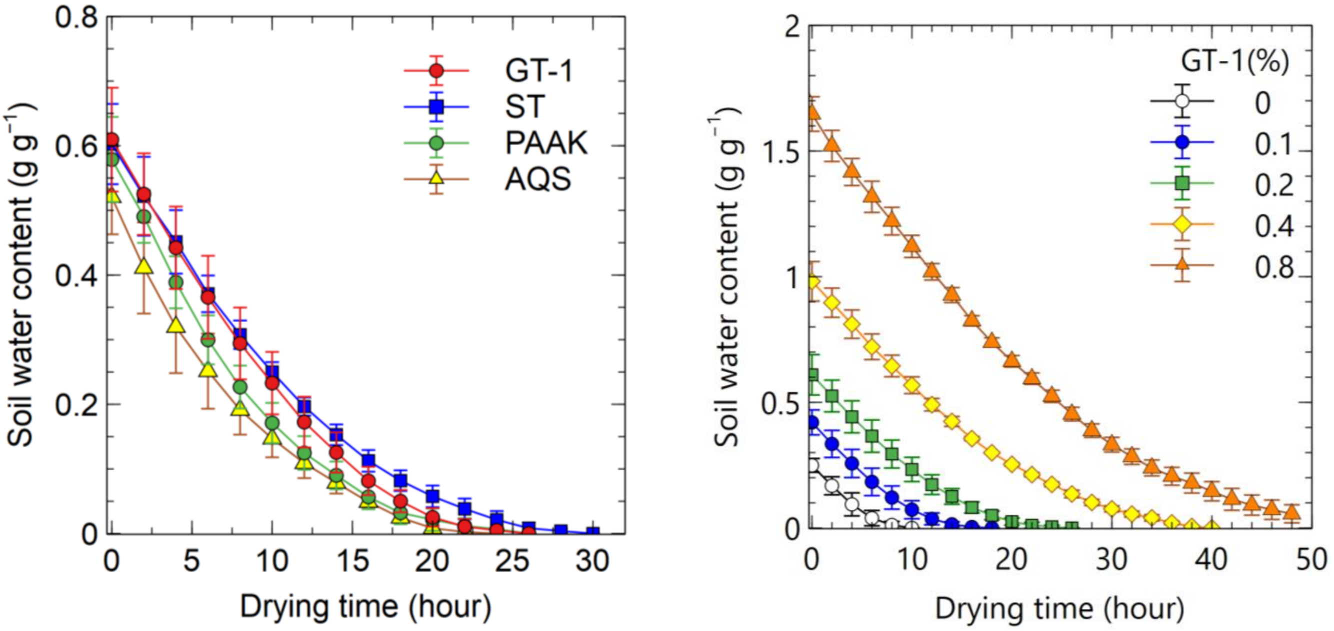


Figure 2. Relationships between the SAP (GT-1) application rate and both the soil water content and the SAPs' water content. Each point shows the mean and standard error (n = 3). The line on the red circles was drawn by regression analysis. Soil water content = 1.823 SAP% + 0.2761, R^2 = 0.9997.

3.2.3. Experiment 5

The soil–SAP mixtures with a rate of 0.2% were completely desiccated in 26.7–30.0 h under heating at 60 °C (Figure 3a and Table 4). In the case of soil without SAP, complete desiccation of the soil required 11.3 h (Table 4). A higher soil water content resulted in a longer time until complete desiccation of the soil–SAP mixtures, with the longest being 58 h required for the soil–0.8% SAP mixture (Figures 3b and A1).



(**a**) (**b**)

Figure 3. Changes in the soil water content of the soil–SAP mixtures during heating at 60 °C. Comparison among SAP types (**a**) and SAP (GT-1) application rates (**b**). Error bars indicate the standard error of the means (n = 3).

The rates of evaporation from the soil–SAP mixtures decreased with decreasing soil water content. The average rates in the first four hours were 0.0378–0.0503 g g^{-1} h^{-1} for all four SAP types, which was not significantly different from that of soil alone (0.0388 g g^{-1} h^{-1}) (Table 4). The evaporation rates remained almost stable as the rate of SAP (GT-1) addition increased, except for the 0.8% SAP rate (Table A2).

3.3. Soil Water Retention of Soil–SAP Mixtures

3.3.1. Experiment 6

The water contents of all four soil–0.2% SAP mixtures were 0.513–0.584 g g^{-1} in the soils and 255–293 g g^{-1} at a water potential of −0.98 kPa (Figure 4 and Table A3). The soil–water retention curves all had similar shapes, with statistically insignificant differences, and the amount of water held in −0.98 to −3.92 kPa, −3.92 to −98.1 kPa, and <−98.1 kPa ranged from 0.298 to 0.375 g g^{-1}, from 0.032 to 0.073 g g^{-1}, and from 0.125 to 0.182 g g^{-1}, respectively, while their proportions of the total amount of water content were 58.2–66.7%, 6.3–13.4%, and 22.6–35.5%, respectively (Table 6). For the amount of plant-available water in a potential range from −3.92 to −98.1 kPa, no significant differences were observed among soils with and without SAP.

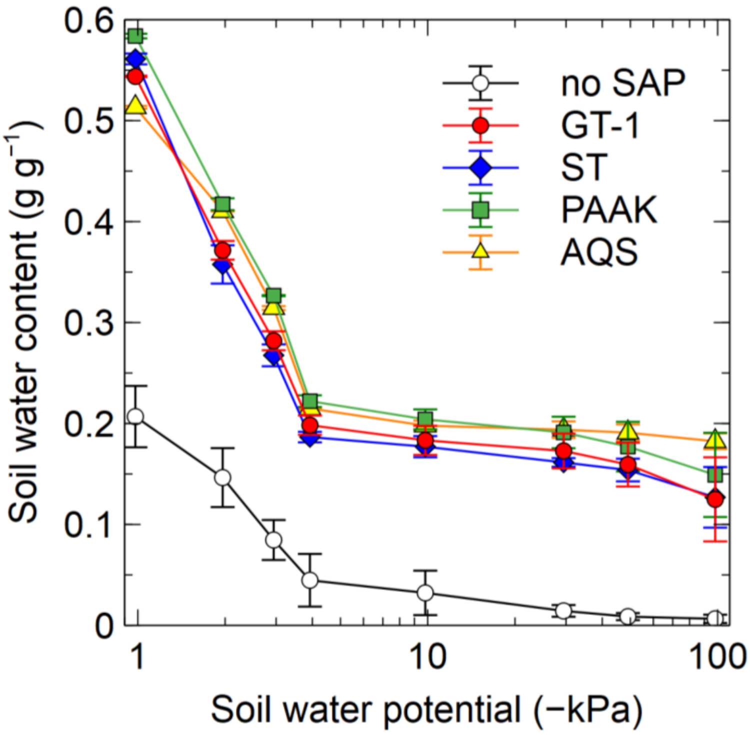


Figure 4. Water retention curves of the soil–0.2% SAP mixtures and the soil without SAP. Error bars indicate the standard error of the means (n = 3).

Table 6. Comparison of the amount of water retained in the soil–SAP mixtures (four SAP types and no SAP) for different potential ranges of −0.98 to −3.92, −3.92 to −98.1, <−98.1 kPa, and the total amount of water at −0.98 kPa. The proportions (%) of retained water to the total amount of water at −0.98 kPa are indicated in square brackets.

SAP	−0.98 to −3.9 kPa	−3.92 to −98.1 kPa	<−98.1 kPa	Total
GT-1	0.345 (0.051) ab [63.5%]	0.073 (0.039) [13.4%]	0.125 (0.004) a [23.0%]	0.543 (0.001) bc [100%]
ST	0.375 (0.000) a [66.7%]	0.060 (0.021) [10.7%]	0.127 (0.026) a [22.6%]	0.562 (0.004) ab [100%]
PAAK	0.362 (0.003) a [62.0%]	0.073 (0.031) [12.5%]	0.149 (0.036) a [25.5%]	0.584 (0.002) a [100%]
AQS	0.298 (0.000) b [58.2%]	0.032 (0.006) [6.3%]	0.182 (0.007) a [35.5%]	0.512 (0.001) c [100%]
No SAP	0.192 (0.036) c [85.4%]	0.032 (0.019) [14.2%]	0.001 (0.000) b [0.4%]	0.225 (0.015) d [100%]

Each value is the mean and the standard error is given in parentheses (n = 3). Within a column, the means followed by the same letter are not significantly different at $p = 0.05$.

3.3.2. Experiment 7

When gel SAP was applied to the soil, the soil–SAP mixtures also swelled in the sand at water potential of −0.98 kPa; the water content was 0.202–0.446 g g^{-1} in the soils and 76.2–158.2 g g^{-1} in the SAP (Figure 5). At first, the soil water content was lower in the soil–gel SAP mixtures with smaller water/SAP ratio, but tended to increase more in the sand at −0.98 kPa. Meanwhile, the water content decreased slightly when the water/SAP ratio was 100 (Figure 5). The shapes of the water retention curves of the soil–gel SAP mixtures were very similar to those for the soil–dry SAP mixtures (Figure 4), but the overall soil water content was significantly lower in soil–gel SAP mixtures than in soil–dry SAP mixtures, even though the SAP application rate was double (0.4%). The soil water content for <−98.1 kPa with the gel SAPs (0.055–0.118 g g^{-1}) was lower than that in soil–dry SAP mixtures (0.125–0.182 g g^{-1}).

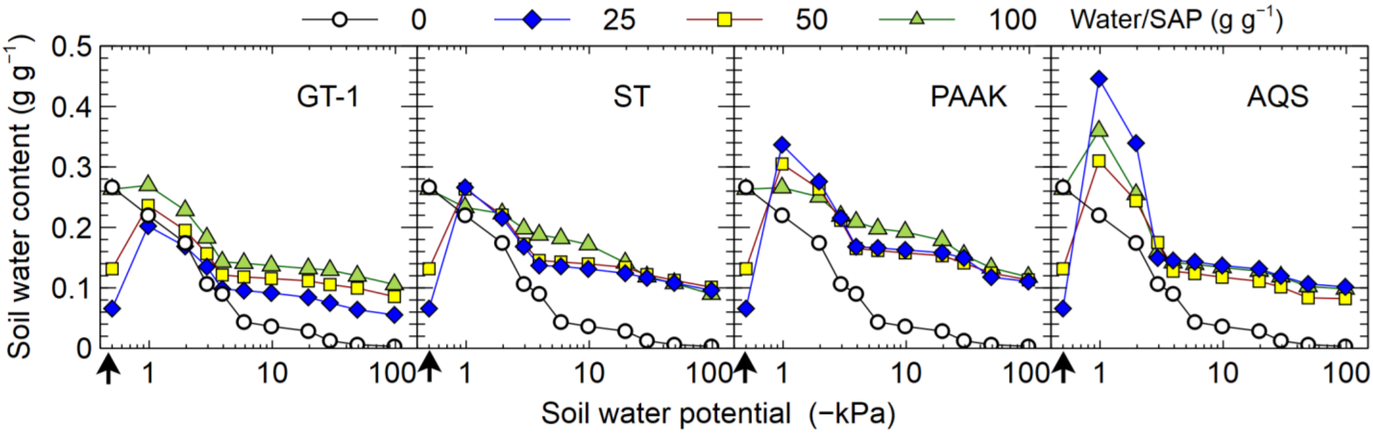
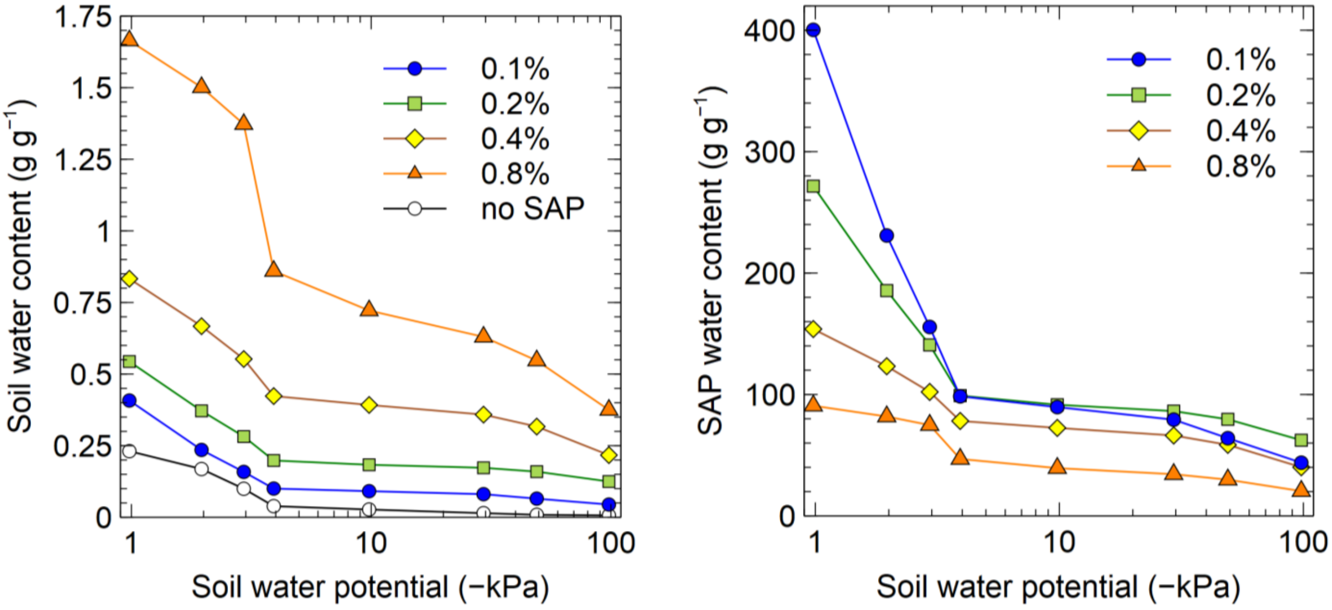


Figure 5. Water retention curves of the soil–SAP (0.4%) mixtures prepared by gel SAPs with different water content. The arrow on the X axis indicates the soil water content at the initial water SAP ratios.

3.3.3. Experiment 8

As the application rate increased, the water retention curves shifted, showing higher water content throughout the ranges of soil water potential (Figure 6a). Overall, the soil water content increased with increasing SAP application rates (Figure A2). Specifically, the amount of water in the range from −0.98 to −3.92 kPa tended to increase at lower SAP application rates, whereas the amount of plant-available water (−3.92 to −98.1 kPa) increased at higher application rates. As for the SAPs' water contents, the soil–0.1%

SAP mixture showed the highest SAP water content (400.3 g g^{-1}) at a water potential of −0.98 kPa (Figure 6b and Table A3), which was almost the maximum water-holding capacity of the SAP. In contrast, the lowest SAP water content (90.7 g g^{-1}) occurred in the soil–0.8% SAP mixture at −0.98 kPa. The proportion of water in the potential range of −0.98 to −3.92 kPa to the total water content was larger when the SAP application rate was lower, such as 75.4% in 0.1% SAP and 48.3% in 0.8% SAP (Table 7).



(**a**) (**b**)

Figure 6. Water retention curves of soil water content (**a**) and SAP water content (**b**) in the soil–dry SAP (GT-1) mixtures with different SAP application rates.

Table 7. The amount of water retained in the soil–SAP mixtures for different potential ranges of −0.98 to −3.92, −3.92 to −98.1, <−98.1 kPa, and the total retained water. The proportions (%) of retained water in the total amount of water at −0.98 kPa are indicated in square brackets.

SAP Rate %	−0.98 to −3.92 kPa	−3.92 to −98.1 kPa	<−98.1 kPa–	Total
0	0.221 [89.7%]	0.017 [7.1%]	0.007 [3.0%]	0.246
0.1	0.307 [75.4%]	0.056 [13.7%]	0.045 [10.9%]	0.408
0.2	0.345 [63.5%]	0.073 [13.5%]	0.125 [23.0%]	0.544
0.4	0.410 [49.2%]	0.206 [24.8%]	0.217 [26.1%]	0.833
0.8	0.805 [48.3%]	0.485 [29.1%]	0.375 [22.5%]	1.664

In regard to the soil–water retention curve of the soil without SAP, the total water content was only 0.246 g g^{-1}, 89.7% of which was gravitational water above −39.2 kPa (Table 7). The soil water content at a water potential of −98.1 kPa was 0.007 g g^{-1}, which is an almost dry soil condition.

The application of SAP resulted in large increases in the water retention, both above −0.98 kPa and below −98.1 kPa, but water increased slightly in the range of −3.92 to −98.1 kPa.

4. Discussion

4.1. SAP Application Rate and the Water Absorbency of SAP in the Soil

As the rate of SAP applied to the soil increased, the soil water content also increased: each additional 0.1% of SAP applied increased the soil water content by 0.183 g g^{-1} on average according to our linear regression analysis (Figure 2). However, the SAPs' water content decreased as the application rate increased. This is probably because the swelling of SAP was confined by the pore spaces in the soil and the volume of the teabag used, indicating that the water absorbency of SAP depends on the space in which the SAP is able to swell. With limited pore spaces, SAPs applied at a low rate swelled fully in the soil, as the porosity of the sand used was 42.6% in this experiment. For better performance in terms of water absorbency, it is recommended to applied SAPs at a low rate. Yu et al. [49] also suggested that application of a low amount of SAP was effective for increasing the water content in the soil. If the water retention in the SAP is insufficient, it would be effective to expand the pore space in the soil for the SAP to swell, e.g., by plowing and adding organic and inorganic materials to the soil [50,51], or the areas of SAP application need to be widened. The teabag method can be used to estimate a suitable rate of applying SAP to the field soil. According to previous studies, 0.2–0.6% SAP is usually adopted empirically or experimentally [52–54]. To determine the most effective SAP application rate, however, the amount of water applicable, the soil's pore volume, the rate of water taken up by plants, and the cost of applying SAP should be comprehensively considered.

4.2. Effects of Salt on the Absorbency of SAPs

The maximum water-holding capacities of the SAPs in pure water varied by about twofold; this might have been influenced by the SAPs' materials and hydrophilicity, cross-linking density, and counterions [22]. Water-holding capacity decreased in salt solutions, especially Ca solutions. Generally, water absorbency decreases in salt solutions of divalent cations and heavy metals [27,30,55]. The negative charges of polymers were neutralized by cations, and the repulsion between charged sites weakened, resulting in shrinkage of the swollen SAP body. As a result, the maximum water-holding capacity of the SAPs did not vary significantly in salt-affected environments. In Experiment 2, the SAPs' water content often decreased over time, suggesting the gradual progression of the cation exchange reaction within the SAP. The ability of SAPs to absorb water needs to be evaluated in terms of the salinity of the soil in the field.

4.3. Water Released from SAP by Heating

In response to heating at 60 °C, the water in the soil–SAP mixtures was released by evaporation, and the evaporation rate gradually decreased as the water content declined. In the first four hours, the evaporation rates were about 0.04 $g\ g^{-1}\ h^{-1}$, irrespective of the SAP type. Basass et al. [56] classified the water evaporation rate of SAPs into the following three stages, with the heat of vaporization also increasing in this order: (1) evaporation of free water, (2) evaporation from water associated with the polymer's molecular chains, and (3) water trapped strongly between molecular chains. More than half the water was associated with Stage 1, and only a small percentage was associated with Stage 3. This corresponds to the evaporation curves in this study. The evaporation rates were almost stable up to the 0.4% SAP application rate (Table A2). In the initial stage, the evaporation rates of free water in the SAPs were almost the same, irrespective of the SAP application rates. This seems to correspond to a large amount of water being retained under high water potential in the water retention curves in Experiment 7 and 8. We are not sure of the reason why the highest evaporation rate occurred with the 0.8% SAP application rate.

4.4. Recovery of Water Absorbency after Desiccation of SAPs

The water absorbency of desiccated soil–SAP mixtures previously immersed in salt solutions was recovered by immersion in tap water, except when the mixtures had been immersed in a 10 mM $CaCl_2$ solution. There are several reports that irrigation water containing salts deteriorates SAPs' water absorbency during wet–dry cycles [34,55,57]. However, it was possible to recover water absorbency by immersing the SAPs in tap water with a low salt concentration. This is probably because the ion exchange with negatively charged sites in the polymers is reversible. This recovery, however, did not occur for SAPs immersed in the 10 mM $CaCl_2$ solution. Bo et al. [55] observed that SAPs' water absorbency was degraded by divalent and trivalent cations and by cations with large ionic radii, probably due to structural damage to the polymers in the SAP. In our experiment, the appearance and texture of the SAPs immersed in the 10 mM $CaCl_2$ solution changed. Since the concentration of 10 mM $CaCl_2$ was only a medium level in terms of the salt concentrations in irrigation water [47], controlling the quality of irrigation water is indispensable for maintaining SAPs' performance. Bai et al. [58] recommended that SAPs should not be dried by more than 80% to maintain their water absorption ability. It may be necessary to monitor and adjust the irrigation to maintain the soil moisture so as to prevent the SAP becoming completely desiccated. Otherwise, the desiccated soil–SAP mixtures could harden, which might be harmful to root growth.

4.5. Difference in the Water Absorbency of Soil–SAP Mixtures between Gel and Dry SAPs

The application of dry SAP to the soil showed greater water absorbency than gel SAPs, even though the application rates of dry SAPs were lower than those of gel SAPs. This trend was unexpected, but held true for all SAP types. The soil water content of the soil–gel SAP mixtures was 0.202–0.446 g g^{-1}, but that in the soil–dry SAP mixtures was 0.513–0.584 g g^{-1} at a water potential of -0.98 kPa. The application of dry SAPs was superior to gel SAPs in terms of water absorbency. The reason for this is uncertain, but the strong hygroscopicity of dry SAP might be a driving factor.

It was noted that the soil–SAP mixtures were able to swell upward without restriction in our experimental conditions. This is unrealistic in the field, except for surface soil. Thick overlying soil layers are usually loaded onto the buried SAP, which restricts the SAP from swelling in the soil [41,59]. Lejcuś et al. [41] indicated that 1 g of SAP with a water absorbance capacity of 200 g could only absorb 5 g of water at a soil depth of 30 cm. Further experimentation is needed to evaluate the dependence of actual water absorbance on the application depth of SAPs in the field. Another application method that directly injects SAP gel into deep soil layers has been reported, but this needs special equipment combining the functions of watering and applying the SAP [10].

4.6. Soil–Water Retention Curves of Soil–SAP Mixtures

The water retention curves of soil–SAP mixtures were similar among all four SAP types. A large amount of water was retained in the range of -0.98 to -3.92 kPa, but this is not usually classified as a plant-available water because it is drained within a short time. The application of the SAP changed the water to a form that is readily available to plants by retarding drainage. It is interesting that abrupt changes in the water content occurred at -3.91 kPa in the water retention curves of all SAP types. A large part of the water in the SAP seemed to exist between swelling cross-linked polymers as almost free water with higher potential and water below -3.91 kPa was probably associated with the polymers in the SAP. Water retained at the potential range between -3.92 and -98.1 kPa is considered to be plant-available water, but only a small increase in the amount of available water occurred: soil–0.2% SAP mixtures held 0.032–0.073 g g^{-1}, which was similar to that in the soil without SAP (0.032 g g^{-1}). The mechanism of the abrupt change in the soil water potential is not certain, but it is considered that the onset of a deficit in the water available to plants grown in SAP-amended soil may be sudden. Regarding soil water below a potential of <-98.1 kPa, although unavailable water below the wilting point (<-1500 kPa) was included, large amounts of water were retained. Considering the fact that the soil without SAP retained only 0.001 g g^{-1} of soil water at <-98.1 kPa, the application of SAP created moist conditions. Together with the findings that a slow evaporation rate occurred for soil–SAP mixtures with a low water content (Figure 3), the moist soil conditions would probably persist. This would presumably change the soil's hydrothermal regimes, such as the specific heat capacity of the soil [9]. Okumura et al. [60] reported that the addition of 0.2% SAP reduced the diurnal range of soil temperature by 2 °C. As an additional effect of SAP, soil that is made moist by SAP might mitigate the heat stress of plants grown in SAP-amended soil.

The actual water retention in the field would be lower than that measured in our laboratory study because of the load from the soils above [40,41]. Thus, it is recommended that a low SAP application rate be used to achieve high performance in SAP-amended soil. Further study is needed to examine the water retention curves of soil–SAP mixtures under the restricted swelling conditions by using the actual field soils to which the SAP has been applied.

4.7. Differences among SAP Types

GT-1, PAAK, and ST are made of polyacrylic acid. When mixed with soil, they showed similar characteristics in terms of water absorbency. The characteristics of AQS are significantly different in terms of several physical parameters. AQS differs from the other SAPs in its larger grains and component polymer materials: it is a copolymer of polyacrylic acid and polyacrylamide. In general, larger SAP grains absorb water more slowly, and their maximum water-holding capacity is lower [23,24]. A relatively high proportion of water was held in the soil–AQS mixture at a water potential of <-98.1 kPa, suggesting that the amount of plant-available water was relatively low. However, when immersed in the 10 mM $CaCl_2$ solution, only the water absorbency of AQS remained unaffected. AQS, with some modifications, may potentially be applied to salt-affected soil as an effective amendment.

5. Conclusions

SAPs can be used as a soil amendment for improving water retention in sandy soils because they impede the quick drainage of water due to their high and rapid water absorption. The original characteristics of pure SAPs were concealed upon mixing with soil. Salt solutions deteriorated the SAPs' water absorption, so it is more effective to use SAPs combined with irrigation with low salt concentrations. Since the SAPs' absorption of water resulted in their swelling, it is important to have sufficient pore space in the soil to ensure performance. The soil layers developed in soil structures may be suitable positions for SAP application. In addition, we found that a lower SAP application rate was more effective for improving the water available to plants. Evaporation from soil–SAP mixtures became slower with decreasing soil water content, with an extension of the time for the complete desiccation of the soil. The application of the teabag method to characterize the SAPs' performance can be recommended as a simple and low-cost tool for soils in the field. Because the soil's water dynamics are affected by the water consumption of living roots in the field, further study is needed with accompanying growing plants in various soil conditions. Furthermore, since SAPs do not generate water and their water absorbency does not last for long, further examinations must confirm the effective period of SAPs' performance, depending on the crop species and soil characteristics under specific water management systems. Methods of applying SAPs in future should be selected according to the costs and benefits in terms of the durability or biodegradability of the SAPs, their handling in the field, and the production of target crops.

Author Contributions: M.T. performed the experiments and wrote the manuscript; S.O. initiated the study and supervised the research; I.K. analyzed water retention curves. All authors have read and agreed to the published version of the manuscript.

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Appendix A

Table A1. Maximum water-holding capacity (MWHC) of four SAP types in pure water, 10 mM KCl, and 5 mM $CaCl_2$ solutions.

SAP	MWHC in Pure Water	MWHC in KCl	MWHC in $CaCl_2$
GT-1	494 (18) a	182 (7) a	114 (3)
ST	441 (23) b	178 (5) ab	87 (17)
PAAK	361 (10) c	167 (2) b	66 (18)
AQS	296 (7) d	136 (3) c	98 (8)

Each value is the mean and the standard error is given in parentheses (n = 3). Within a column, means followed by the same letter do not differ significantly at $p = 0.05$.

Table A2. Comparisons of soil water and SAP water contents, time to desiccation, and the initial (0–4 h) evaporation rate at 60 °C from the soil–SAP mixtures.

SAP	Soil Water Content (g g^{-1})		SAP Water Content (g g^{-1})	Time to Desiccation (hour)	Evaporation Rate (g g^{-1} h^{-1})
	3 h	24 h	24 h		
GT-1	0.751 (0.018) a	0.665 (0.067) a	325 (19)	28.7 (0.7) a	0.0418 (0.0043)
ST	0.755 (0.012) a	0.671 (0.051) a	330 (15)	30.0 (1.2) a	0.0378 (0.0060)
PAAK	0.740 (0.012) a	0.650 (0.041) a	322 (12)	26.7 (1.8) a	0.0475 (0.0078)
AQS	0.566 (0.017) b	0.593 (0.018) a	293 (5)	28.0 (1.2) a	0.0503 (0.0044)
no SAP	0.308 (0.012) c	0.299 (0.006) b	n.a.	11.3 (2.2) b	0.0388 (0.0106)

Each value is the mean and the standard error is given in parenthesis (n = 3). n.a. is not applicable. Within a column, means followed by the same letter do not differ significantly at $p = 0.05$.

Table A3. SAP water contents (g g^{-1}) of the soil–dry SAP mixtures in the water potential range of −0.98 to −98.1 kPa.

SAP Type	SAP Application Rate (%)	Potential (kPa)	−0.98	−1.96	−2.94	−3.92	−9.81	−29.4	−49.0	−98.1
		pF	pF1.0	pF1.3	pF1.5	pF1.6	pF2.0	pF2.5	pF2.7	pF3.0
GT-1	0.1		400.3	230.9	155.6	98.5	89.6	79.3	64.0	43.8
GT-1	0.2		271.6	185.6	140.8	99.0	91.5	86.3	79.5	62.3
GT-1	0.4		153.9	123.3	102.1	78.2	72.5	66.2	58.5	40.1
GT-1	0.8		90.7	81.8	74.8	46.9	39.3	34.3	29.8	20.4
ST	0.2		278.0	177.0	132.5	92.4	87.6	79.9	76.2	62.6
PAAK	0.2		293.3	209.6	164.1	111.6	102.5	96.0	89.0	74.9
AQS	0.2		255.0	203.9	156.2	106.8	98.3	96.3	94.8	90.6

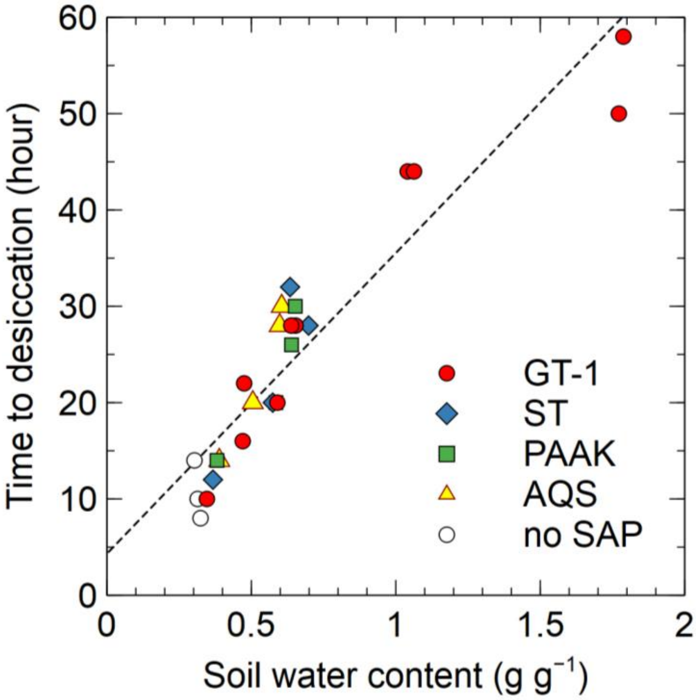

Figure A1. Relationship between the soil water content of soil–SAP mixtures and time to desiccation by heating. The dotted line was drawn using the regression analysis y = 31.0x + 4.7, R^2 = 0.8687.

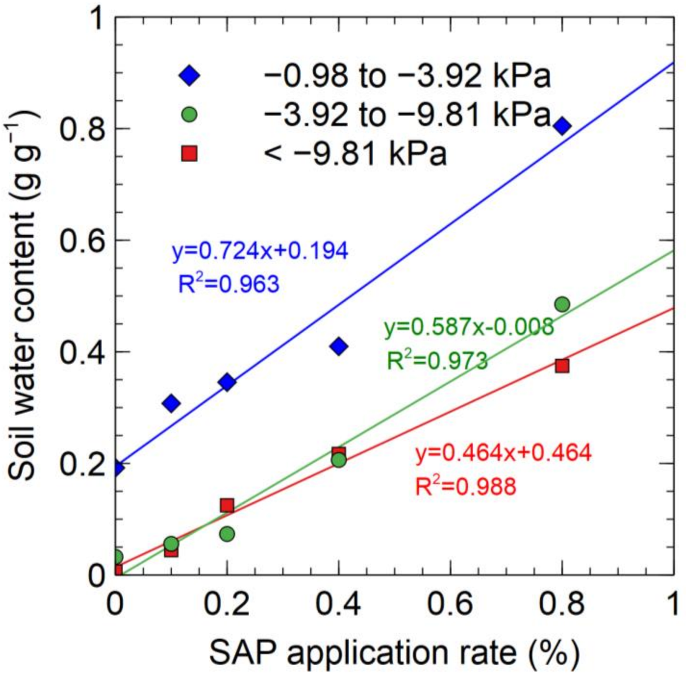

Figure A2. Relationship between the SAP application rate and the soil water content of each range of water potential. The colored lines and equations are regression analyses of the symbols with the same colors.

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