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Super Absorbent Polymers (SAPs)

A Final year project report submitted in partial fulfillment of the requirements for the degree of bachelor of science of polymers and Petrochemical Industries in the faculty of Materials Engineering/Babylon University

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A.D 2023 **1444 A.H**

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ٱللَّهُ لَآ إِلَٰهَ إِلَّا هُوَ

ٱلْحَىُّ ٱلْقَيُّومُ ۚ لَا تَأْخُذُهُۥ سِنَةٌ وَلَا نَوْمٌ ۚ لَّهُۥ مَا فِى ٱلسَّمَٰوَٰتِ وَمَا
فِى ٱلْأَرْضِ ۗ مَن ذَا ٱلَّذِى يَشْفَعُ عِندَهُۥٓ إِلَّا بِإِذْنِهِۦ ۚ يَعْلَمُ مَا بَيْنَ
أَيْدِيهِمْ وَمَا خَلْفَهُمْ ۖ وَلَا يُحِيطُونَ بِشَىْءٍ مِّنْ عِلْمِهِۦٓ إِلَّا بِمَا
شَآءَ ۚ وَسِعَ كُرْسِيُّهُ ٱلسَّمَٰوَٰتِ وَٱلْأَرْضَ ۖ وَلَا يَـُٔودُهُۥ حِفْظُهُمَا ۚ
وَهُوَ ٱلْعَلِىُّ ٱلْعَظِيمُ ٢٥٥

Dedication

To the one whose name I carry with pride.....

To the one who sought to enjoy comfort and contentment...

To the one who was stingy with nothing to push me towards the path of success...

To my dear father
To the inexhaustible fountain...

To the meaning of tenderness, love and devotion...

To the one whose supplication was the secret of my success......

To the most precious person in existence...

To my dear mother
To those who embodied love in all its meanings....

to support and giving...

to those who were my refuge...

to those who were closer to me than my soul...

to my dear brothers...

To the one who lines the lines of life with me...

to my life partner and companion...

to my dear husband
To the companions of the path.....

To those who forgot me in my studies......

To my loyal friends

Thanks and appreciation

To the College of Materials Engineering, Department of Polymer and Petrochemical Industries, for their efforts to make engineers with the utmost understanding and awareness, to the doctors who established the foundation of engineering in us, to all the workers in the Polymer Department, including two teachers and professors, to the source of knowledge and the tree of sincerity, Prof. Dr. Adua Jabbar Braihi, who I am proud to be one of his students, thank him for His chest capacity, abundance of knowledge, and his constant and diligent follow-up to me to reach the best results And big thanks to the professors in the lab for helping me in all practical aspects

Abstract

In the current work, an agricultural grade of Super Absorbent Polymer (SAP) used to monitor its effects upon the growth of Cress and Green Beans plants. For Cress plant, SAP was mixed by 0, 5 and 10 wt% with soil and for Green Beans mixed by 0, 5, 10, 15 and 20 wt%.

SAP analysis results showed that it is belongs to Acrylic family and have sufficient mechanical and thermal properties, but losses about 29% of its initial weight during swelling and have low retention ability.

Results showed that it is neutral polymer and can used with acidic and alkaline environments. Its absorbance capacities with both ordinary water and saline solution, refers to possibility of used it in various lands types because of the comparable values (34.09 g/g vs. 30.683 g/g).

The absorbance kinetics results showed similar behaviors with ordinary water and saline solution but with less values in saline conditions.

Weight and length of the Cress plant increased linearly with the SAP content but the 5 wt% SAP gives maximum number of branches of the plant, better vegetation, better density and best color intensity of the leaves.

Results of Green Beans showed that a positive effects of on the growth ability, color intensity, coverage percentage and flowers number.

CHAPTER ONE

INTRODUCTION & LITERATURE REVIEW

1.1 SAP definition

Super absorbent polymers (SAPs) is a type of polymer that has the ability to absorb more liquids20% of its original weight without disintegration due to the cross-linking between its chains. Therefore, its uses varied in agriculture (to rationalize the consumption of irrigation water and retain excess rain water) and in industry such as wastewater treatment and adsorption of heavy metal ions and artificial snow. It is also used in the medical field as drug delivery systems and many more.

A superabsorbent polymer is hydrophilic, water-absorbing polymers or copolymers that can absorb and retain very large amounts of liquid relative to its mass. Water absorbent polymers, which are classified as hydrogels when mixed, aqueous solutions through hydrogen bonds with water molecules.

1.2 How it absorbs water

They are called polymers “SAP” and hydrogel, and absorbent polymers, Absorbent gels, Super soaks, super slimes, And gel water, It is considered kind of a synthetic macromolecular polymer that absorbs water, As it has the ability to absorb 100,000 of the weight of the existing water, And in a short period, by absorbing it and making it in the form of granules that remain in the soil to supply it with what is needed, Where it plays its role as a hygroscopic material that takes the shape of its granules and its color is like white sugar, and it swells after absorbing water to become a gel. A clearly, It maintains the same level of humidity inside, even when subjected to pressure, It does not explode or rupture.

1.3 Literature Review of Agricultural Applications

Superabsorbent polymers have many applications, including in agriculture, and many countries used these polymers in agriculture and conducted many experiments, and they were successful. Some of these countries used superabsorbent polymers in agriculture:

1.3.1Iraq

1-In Anbar, an experiment was conducted to apply superabsorbent polymers in agriculture, where researchers at the university conducted the experiment on maize and wheat seeds.(Wheat) where these seeds were sown in plastic containers used for the purpose of agriculture and after germination, polymer crystals were placed in seedling containers near the root and left for four days without watering. Compared to seedlings that contain a polymer, where the seedlings added to the polymer were not affected by the delay in watering, which indicates that the soil remains moist, retains water in it, and provides it to the seedlings in the event of no watering.

1-3-2Egypt

2-A study of farmers' experiences in the Studies and Research Complex of the tourist city of Abu SimbelToshka - Aswan - Egypt in the winter season 2013/2014,2014/2015. Wheat crop was used in the study in the general section,Where the drawing was studied in the section that was modified in the department section. A study of the extent of cultivar wheat yield (Egypt 1,Egypt 2) subjected to stress for the entire period of its growth in addition to the treatment with superabsorbent polyurethane (hydrogel) full length cultivation in the soil. The image also appears in the form of the grain yield in the spike, the weight of the crops, the weight of the grain, the weight of the grain, the weight of 1000 grains, and the date of expulsion of the spikes.,Egypt 2) Results of different irrigation formulas (60-80-100-120%) hydrogel). General recommendations: - The highest yield of wheat crop for Egypt 1 cultivar,Egypt 2 was when using the ratio (120%) of the total productivity of production,or productivity,Or production regulations (hydrogel). - The less productive wheat crop,Egypt 2,It was when using (60%) of the suitability of plants,and buildingaOn these papers (60%) effective for saving water. It must be taken into account as a result of facing a particular crisis,and buildingaon certain results,The result of producing certain results in certain results,by 20% in resolution outcomes. Toward savingsaof the land and achieve the equation. Experimentation with questions (it was a real study in the first phase of the first season). Its growth in addition to treatment with absorbent polymers (hydrogel). In the first season 2013/2014, the treatment was applied between the different irrigation treatments 60-80-100-120 of the needs. The results indicate that the results are specific for all the different irrigation treatments for my class (Egypt 1,Egypt 2). The experience of the second season 2014/2015 was applied adding another factor in addition to the basis that was conducted in the previous season. To confirm that the results, knowing that the results proved that the results were significant, not only other transactions, but also after adding another factor in the experiment, which is the hydrogel.

1-3-3India

3- The Indian Agricultural Research Institute,In New Delhi, it manufactures a polymer with an improved ability to absorb, called “Buddha Hydrogel”, in order to provide the water required for agriculture.,This is through an absorption capacity based on a natural polymer and a potassium polyacrylate binding agent,Which works to absorb water in severe and semi-arid soil conditions,That is, at a temperature of 40 to 50 degrees Celsius,Indeed, it showed successaA big oneaa,Its ability to absorb 400 times its normal weight,It releases water gradually,Decomposition does not begin until after a year of being in the soil,It is not affected by salts significantly,He worked to reduce soil infiltration with fertilizers and pesticides,Raising the percentage of physical properties in the soil, which increased the speed of plant growth and the productivity of seedlings.

1-3-4China

4- In northern China Oat is an important grain and forage crop and is now being cultivated as a promising forage crop in northern China. Increased land degradation and shortage of forage resources for animal production during winter have accentuated the need for alterative forage crop such as oats. Low precipitation, poor soil quality and cool temperatures are major limitations to cereal grain production in the northern regions of China Early sowing of oat in the area begins in early May and late sowing is early to mid July. Due to the short growing season (frost free period), most farmers grow out in the early season. Considering the strong wind during winter, it is also important to provide a surface soil cover

Soil erosion is also a major environmental and agricultural problem in northern China. Song et al. "has indicated that out can provide protective cover over the soil surface and reduce wind) erosion in the area. For the same wind velocity, the anti-erosion capacity was highest in oat stubble land (53.3%) followed by maize (17.8%) , sunflower (11.1%) and mung bean (8.9%) in the same area.

Due to shortage of surface water, forage oat cultivation in northern China is almost rain dependent. Sandy soils in the area are characterized by low water-holding capacity, high evapotranspiration and excessive leaching of the scanty rainfall. leading to poor water and fertilizer use efficiency by crops. The problem of inefficient use of rain and irrigation water by crops is most important in semiarid and arid regions
linked polymer developed to retain water in the agricultural and horticultural sector. Earlier, polymers were not used in the agricultural field due to their high prices. Recently, many polymer industries developed around northern China and the prices became comparatively cheaper (about 10 USD/kg). These polymers can also retain soil moisture up to 3-5 years after application.

Thus, the application of SAP in agricultural field has become a popular water-saving technology for many farmers in arid and semi arid regions of northern China. The main objective of this study was to evaluate the effectiveness of different rates of superabsorbent polymer (0, 30, 60, 90 and 120 kg ha') on growth, biomass production, grain yield, and quality of a forage oat (Baiyan 7) in an erosion-prone arid sandy field in northern China.

1-3-5Syria

5- In Syria, a researcher presented an experiment on the application of superabsorbent polymers in agriculture, where he used polymers in the cultivation of A-tomatoes, B-beans, and the experiment was as Addition of superabsorbent polymers to the soil as an effective improving agent and study the effect of its addition on the cultivation of different plants such as beans, tomatoes and peppers

A-Study of bean samples

We tested the addition of the prepared superabsorbent polymeric materials as a soil improver. The composite material was added to the soil at the rate of 0.1% by weight. We show in the beneficial and positive effect of the composite material on the growth of the bean plant, eighteen weeks after the start of cultivation. The

composite material had a zenoei on plant growth. The number of samples in each group is 5 samples.

The results obtained to test the effect of adding superabsorbent polymeric materials to the soil on the growth of samples of bean plants all the results we obtained when studying the effect of adding superabsorbent polymeric materials to the soil when studying samples of the leguminous plant. Sample groups from 1 to 4 are samples added The super absorbent hydrogels prepared in the second chapter with different compositions and with different preparation treatments and at the percentage of 0.1% .

results obtained for testing the effect of adding superabsorbent polymeric materials to the soil on plant growth Beans and their productivity. we can extrapolate the encouraging results that we obtained in the field after treating the soil by adding super absorbent polymeric materials to it. Which can be summarized through the following points: An increase in the growth strength of the treated bean plants compared with the growth of the control plants. An increase in the average number of branches as well as in the length of the largest stem compared to control plants. The average thickness of the largest stem increased compared to the control plants. The average number of bean pods increased compared to control plants...

The addition of the super absorbent hydrogel to the soil made it play the role of a regulator of water. This appears when watering the samples; The samples to which the super absorbent hydrogel was added needed less watering and took as much water as they needed, while the water accumulated in the control plants.

Altogether, we had an increase in the amount of beans produced by two or more times compared to the control plants. In the following table, we show an encouraging, clear and clear improvement in bean production compared to control plants.

The encouraging and clear improvement in the amount of production of bean samples treated by adding hydrogels to the soil comperted with the production of control plants 18 weeks after the start of cultivation.

-B-

Agricultural applications of superabsorbent polymeric materials
Agricultural application on samples of tomato plants

We also tested the effectiveness of the effect of adding superabsorbent polymeric materials prepared as soil improvement material on tomato samples, the composite material was added to the soil at a rate of 0.1% by weight. Eighteen weeks from the start of cultivation, the groups of samples from until they were cultivated by adding the composite material to the soil, while the samples 0 were left as a control. It can be clearly

1- set of control plants...
2- plant group NaAlg-g-P(AA-co-AM) superabsorbent hydrogel was added

presents the totality of the results that we obtained when studying the effect of adding superabsorbent polymeric materials to the soil when studying samples of tomato plants. the sample groups The prepared superabsorbent hydrogels were added to it.

results obtained for testing the effect of adding superabsorbent polymeric materials to the soil when studying samples of tomato plants. the sample groups 3

The prepared superabsorbent hydrogels were added to it

Here, too, we record the encouraging results obtained by adding superabsorbent polymers as a soil improver
In the case of the tomato plant, we show this improvement through the following points:
Witness rapid growth compared with a flowering speed.
Fruiting speed.

The amount of fruiting (productivity).

With the progression of the growth stages: the control plants were mostly smaller compared to the copolymer and materials
The prepared superabsorbent hydrogel.

There was a heavy overlap and intertwining between the plants in the samples to which the prepared superabsorbent hydrogel as for the control plants, there is no overlap.

The separations between the cultivated tandoori plants are quite clear.

Percentage of improvement in the production of tomato plants when

adding the prepared superabsorbent hydrogels compared with control plants 14%

1-3-6 Iran

6-Regarding the large amount of Iranian regions occupied by arid and semi-arid climates, the creation, maintenance and expansion of green spaces faced severe water resources restrictions 30 years ago, a program titled Xeriscaping was created due to the efficient water consumption of green spaces in arid and semi-arid regions. The second principle of this program refers to the physical modification of the soil.

One of the soil amendment materials is the super absorbent polymer. The first applications of these materials in agriculture around the world date back to the 1960s in Iran. However, the use of these materials. In agriculture and green spaces in particular, it is a new phenomenon. Based on the results of a study conducted to evaluate the effects of 5 superabsorbent levels on grass in Tehran (Iran). It is shown that the material increased color intensity, density, coverage area, and reduced fading rate furthermore. It was reported that the most efficient amount was 100 grams per 1 million square meters (Khoshnevis 2006.) according

to the estimated results of fumigation pans in a part of Tehran (Iran). Every 1 m of grass requires 14 to 18 liters of water in warm seasons per day. Saving this amount of water is very difficult. Application of 100g of supersorbent in the said area can reduce water consumption by 50% (Ataei and Ghorbani 2001.) Another study on turfgrass indicated that application of 8g of supersorbent per 1kg of soil enhanced the available moisture up to 4.2, 1.8 and 2.2 times in sandy loamy soils. and loamy and silty, respectively, in the suction range of 0.3 and 15 bar (Musavinia and Atapor, 2006.).

CHAPTER TWO

THEORETICAL PART

2-1 Introduction

Superabsorbent polymers (SAPs) represent a special polymeric materials which can appear in a gel state as a result of absorbing huge amounts of water and aqueous solutions [1]. There are some requirements for polymeric material to quantify as a SAP, such as:

1- Dry polymeric substances must absorb twenty times or more of their original weights from water, saline, or physiological solutions.

2- The swollen hydrogels must kept their initial shapes.

3- The resulting hydrogel should have enough physical integrity to resist flow as well as incorporation with the adjoining particles [2].

4- Retain the absorbed solutions under some pressure [3].

SAPs can absorbs deionized water up to 100,000% (1000 g/g) while absorption capacities for the ordinary hydrogels is not exceeds 100% (1g/g).This ultrahigh absorption capacity makes SAPs ideal for many absorbing applications like disposable diapers, holding soil moisture, adults incontinence pads, medicine for drug delivery systems, bandages to absorb surgical fluids, and controlled release medium [2]**.** Also, these high water contents, give the resultant hydrogels a flexibility level comparable to that of the natural tissue.

SAPs, consist generally from polymeric chains network which are crosslinked to overcome the dissolution tendency and to support water diffusion into the network; there are ionic functional groups available along the chains [4]. SAPs can hold over the absorbed solutions under some pressure due to their loosely cross-linked structures as well as their hydrophilic nature, where the hydrophilic groups are hydrated in an aqueous environment, thus creating a hydrogel structure [5].

The desired lineaments of SAPs involve high capacities of swelling, high rates of swelling, highest durability during storage, as well as good gel strength. Indeed, SAPs cannot fulfill all the desired features, therefore the reaction variables must be optimized to obtain a suitable balance among properties. For instance, hygienic hydrogels should have highest absorption rate, as well as lowest both residual monomer content and rewetting, while an agricultural hydrogel must have higher AUL and lowest sensitivity to salinity [7].

Some of the resultant hydrogels responds to the external stimuli, like pH, heat, electric field, as well as chemical environments. Such responsive hydrogels (called "intelligent" or "smart" hydrogels) attracted an increased attention in many areas like the pharmacy, biotechnology, drug delivery systems, separation processes and agriculture [6].

2-2 Types of Superabsorbent Polymers

SAPs can be classified with different point of view (Figure 1).

1- According to their morphological structures, SAPs can be classified as powders, particles, fibers, spherical, emulsions and membranes. These morphological structures were designed to satisfy the various applications needs.

For instance, sanitary napkins and diapers can be formed by putting the powder product in a multi-layers sheet, while for deodorant application, spherical products and particle can be used. Antistatic electric fibers were manufactured from fiber forms, while membranes utilized in anti-fost sheets. For soaking and painting, an emulsion forms can be used.

2- Based on their resources, SAPs can be categorized to:

a- Natural macromolecules such as polysaccharide-based and polypeptide-based materials.

Polysaccharide-Based SAPs such as cellulose, starch, chitin, chitosan and natural gums (xanthan, guar, and alginates) can prepared by either direct cross-linking of polysaccharide or by graft copolymerization reaction of vinyl monomer (s) upon the polysaccharide in the existence of the crosslinker agent [9].

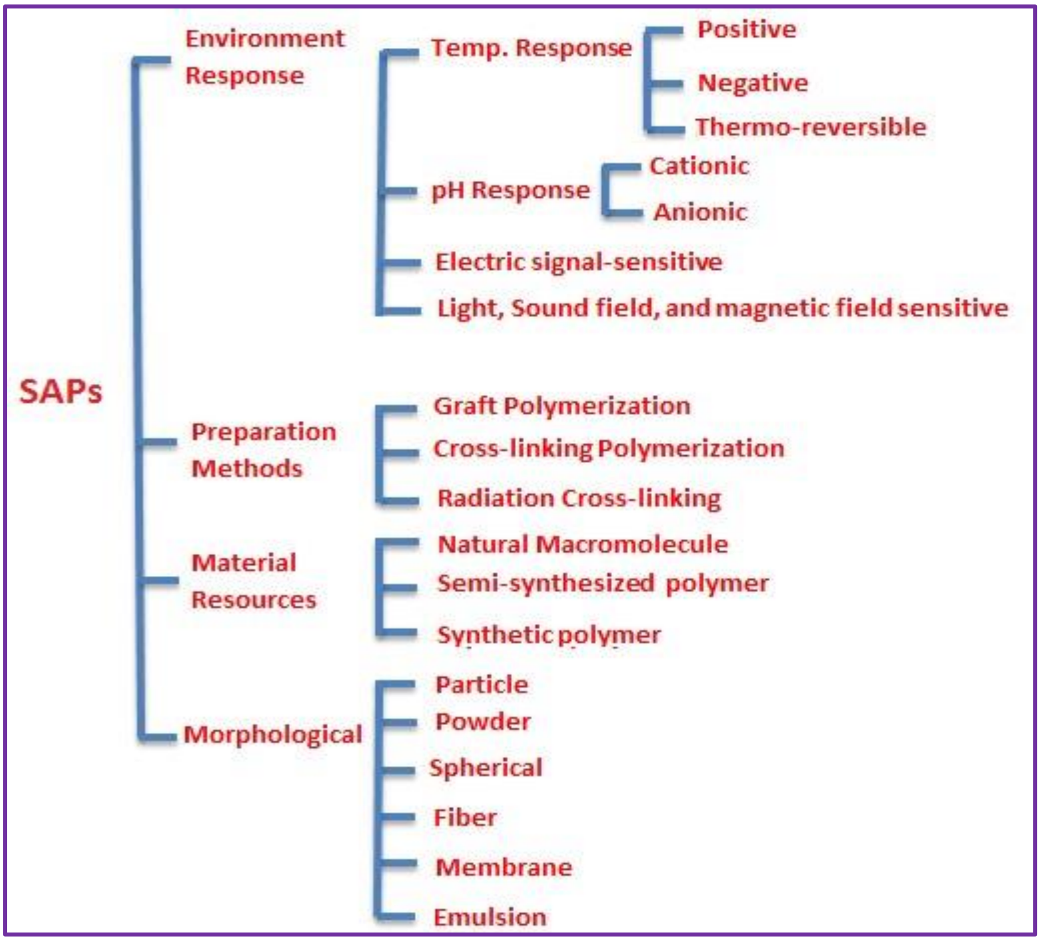

Figure 1: Types of SAPs depended on different point of view [8].

b- Semi-synthesized polymer

c-Synthesized polymers such as petrochemical-based.

As compared with their artificial counterparts, natural-based SAPs possess some advantages like biodegradability (thus escaping the environment from pollution), biocompatibility, their natural source, nontoxic materials, as well as low cost [1]. Furthermore, petrochemical-based SAPs have faced some problems such as increasing of global oil price and pollution concerns. The disadvantage of the natural polymers is the low solubility in water, thus, they are modified chemically to soluble derivatives, such as carboxymethyl starch (CMS) and carboxymethyl cellulose (CMC) [10].

3- Based on preparation method types, SAPs can be divided to crosslinking polymerization, radiation cross-linking, graft polymerization, and networks formation of water-soluble polymer [11].

4- Based on the probability of existence of electrical charges upon the cross-linked chains and types of this charge, SAPs may be grouped into: **a-** Non-ionic polymers:

They absorb water and aqueous fluids via a mixing effect due to the hydrophilic groups. Water molecules are solvated through hydrogen bonds results in a swollen, soft gel [12].

b-Ionic polymers: They contain anionic and cationic polymers. The presence of charges along the backbone greatly increases swelling due to the strong backbone ion-dipole interaction and due to the enhancement of the osmotic pressure in the gel because of the solvation of the counter-ions. **c-** Ampholytic (amphoteric) includes both basic and acidic groups.

d- Polybetaines(zwitterionic) contain both cationic and anionic groups in every repeating unit [4]. Photo-chromic hydrogels, recently were prepared with 2800 g/g WAC.

2-3 Design Considerations

A SAP material involves two processes; swelling and cross-linking.

2-3-1. Swelling Process

Swelling behavior is affected by several factors, such as:

1-Interaction nature: There are four prime forces affect the swelling capacities for the ionic hydrogels: interactions between polymer and solvent, ionic interaction, elastic interaction, as well as electrostatic repulsion. The most important one of these forces is the ionic interaction because of the presence of the mobile ions like K^+ and Na^+ within the SAP structure.

2-Polarity: Generally, for natural polymers there is a one-to-one correlation between the number of polar groups and the number of water molecules adsorbed [14].

3-Elastic retroactive force: Swelling tendency will stop at an equilibrium point when the swelling force is balanced by the retroactive force, which is developed as the chains between the cross-links become increasingly elongated [15].

4-Type and degree of cross-linking: The cross-linker varies the polymeric chain length. Smaller chains have more polymer ends, therefore do not contribute to water absorption, while longer chains have more network space and thus increase swelling.

According to Flory equation (Equation 1), for a given polymer-solvent system the equilibrium swelling (S_e) is a function of the cross-link density

$$S_e^{5/3} = B\, M_c / (1-2M_c / M_w) \quad \text{……………………………………………(1)}$$

Where: B represents a constant for a given polymer-solvent system, M_w is the weight average molecular weight of the similar uncross-linked polymer, M_c is the average molecular weight per cross-link.

5- Solvent properties: Since the swelling involves diffusion of the solvent molecules within the gel network structure, the concentration of the solvent has announced effect according to the Fick's diffusion equation [15]:

$\partial c/\partial t = \nabla (D\nabla c)$………………… ………………………………….…..(2)

Where D is the diffusion coefficient and c is the solvent concentration. 6- Morphological properties (porosity, particle size): the smaller the average grain sizes, the larger the swelling.

7- Conditions of the swelling environment such as pH, temperature, ionic strength as well as the counter ion and its valance [16].

The mechanism of the swelling process contains the following actions: solvent molecules penetrate the polymeric networks structures. Simultaneously, the molecular chains among the cross-linked points expanding, therefore reducing their enthalpy values. This new molecular network structure has an elastic contractive forces which increases the contraction tendencies of the networks (Figure 2).

Unless these two opposed forces get their balance state, expansion and contraction tendencies cannot reach their equilibrium also. So, the osmotic pressure will be the driving force behind the swelling expansion, while the elastic force within the network is responsible of the gel contraction [11].

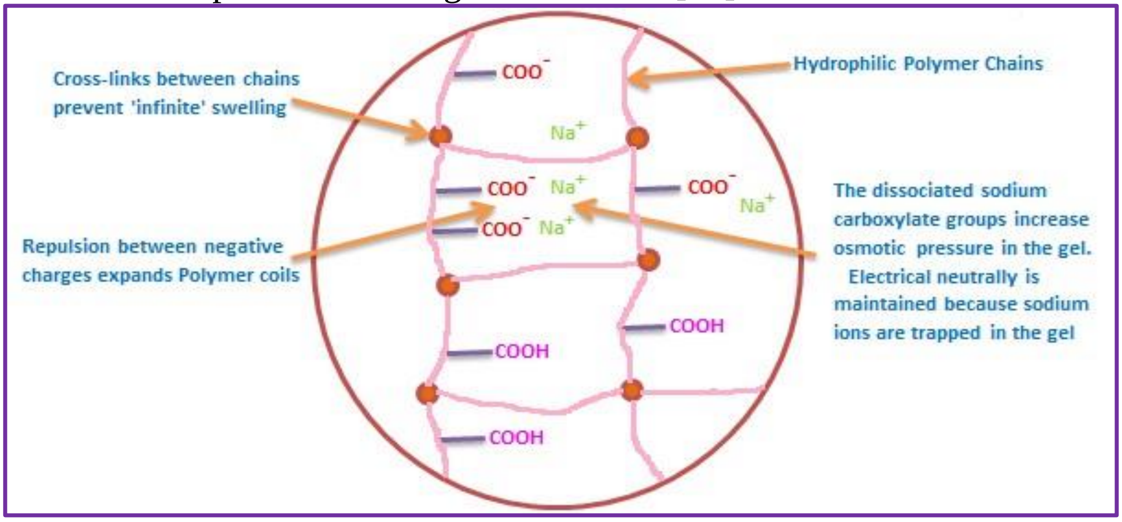

Figure 2: Mechanism of swelling in SAPs [14]

If water is added to the SAP particles, a water /polymer interaction will arises due to the (Mixing Effects), which consists of effects of both hydration(solvation) and the formation of hydrogen bonds.

The hydration is the interaction between solute ions and solvent molecules, i.e. Na^{+} and COO^{-} ions attract the polar water molecules (Figure 3).

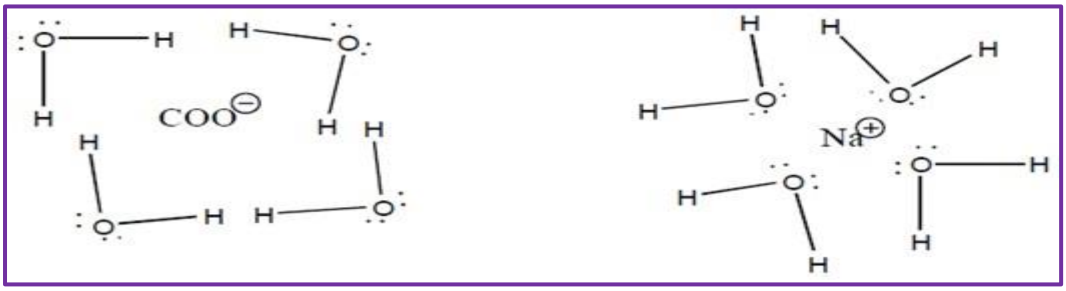

Figure 3: The hydration (solvation) effect [2]

Hydrogen bond (H-bond) which is an electrostatic interaction among molecules, arises in molecules which possess hydrogen atoms connected to atoms with low electronegative like N, F, and O (Figure 4).

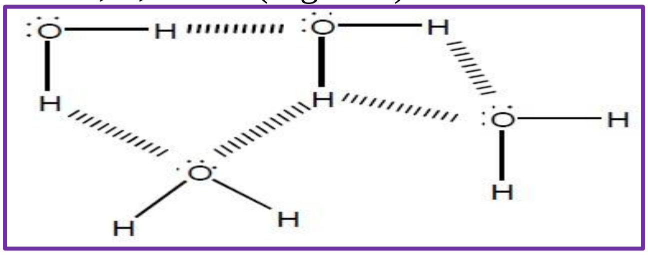

Figure 4:Hydrogen Bonds [2]

These effects creates a cellular structure within the SAP network which can store water molecules.

2-3-2Cross-linking process

Cross-links between polymer chains result in a 3D network; makes SAPs insoluble in water by preventing the polymer to swells to infinity; preventing dissolving. Such behavior arises the elastic retraction forces of the polymeric network. These forces are associated by a reduction of entropy of the chains, since these chains become stiffer from their initial coiled state as shown in Figure 5.

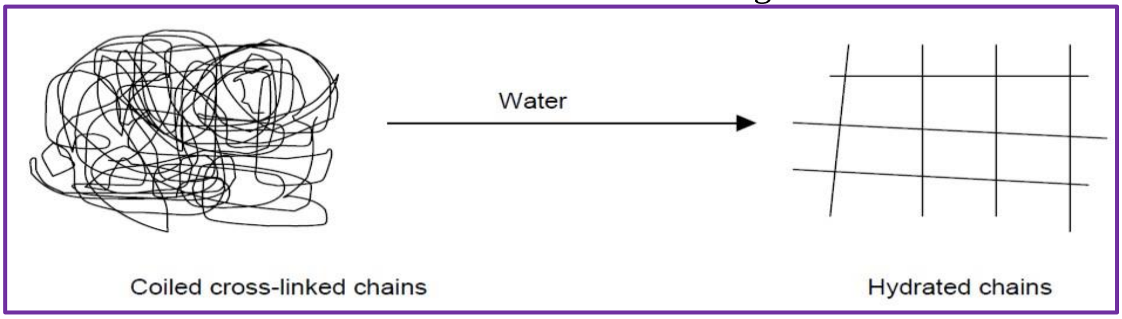

Figure 5: Elastic retraction effect of the network in SAPs [2]

Now, there is an equilibrium between the retraction forces and the chains tendency to swell to infinite dilution. Cross-linking level possess a direct influence on both the swelling capacity range and the gel strength [2]. Several parameters affected the cross-linking efficiency such as, the steric hindrance in the position of the pendant double bonds, solubility level of the cross-linker in the reactants mixture and ability of the cross-linker to exhibit intermolecular addition reactions [7]. Cross-links formed either by covalent, ionic, or H-bonds.

Covalent cross-links are formed either when the major monomers (e.g., acrylic acid) is copolymerized with a di-, tri-, or tetra – vinyl monomer or by reacting the polymer chains with a di- or tri - functional reagents that reacts with the carboxylic acid groups[17].

The reaction between charged polymer chains and polyvalent ion with opposite charge leads to the ionic cross-linking as shown in Figure 6.

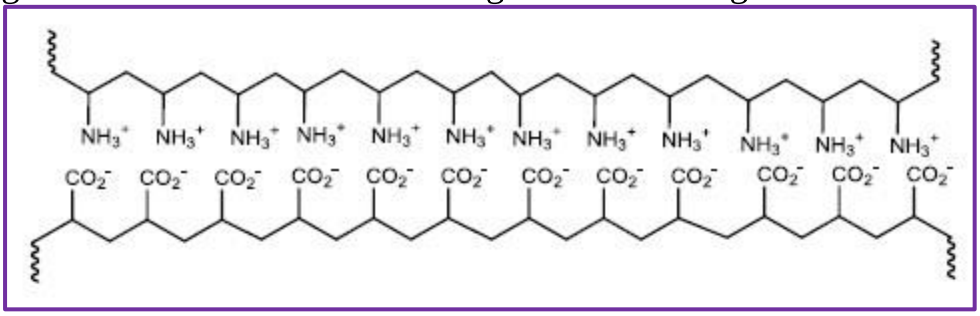

Figure 6: Ionic cross-linking [12]

The creation of H- bonds between segment of one chain with the segment of another chain resulted in the weak chemical cross-linking (Figure 7).

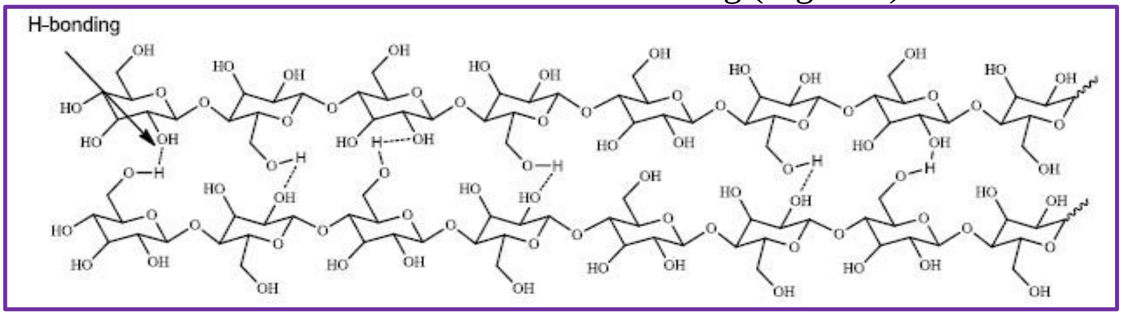

Figure 7: Hydrogen bonds cross-linking [12]

Cross-linking in SAPs, can be classified also into bulk (core) and surface cross-linking. Core cross-linking is usually occurs during the polymerization reaction stage of SAP production [2].

Surface cross-linking of SAP particles can improves both the flow and the absorption against pressure greatly [18]. Particles with surface-crosslinking protected their shapes during the swelling process. This leads to a less densely packed gel bed with air pockets, therefore the fluid can easily flow in high permeability pattern (Figure 8 b).

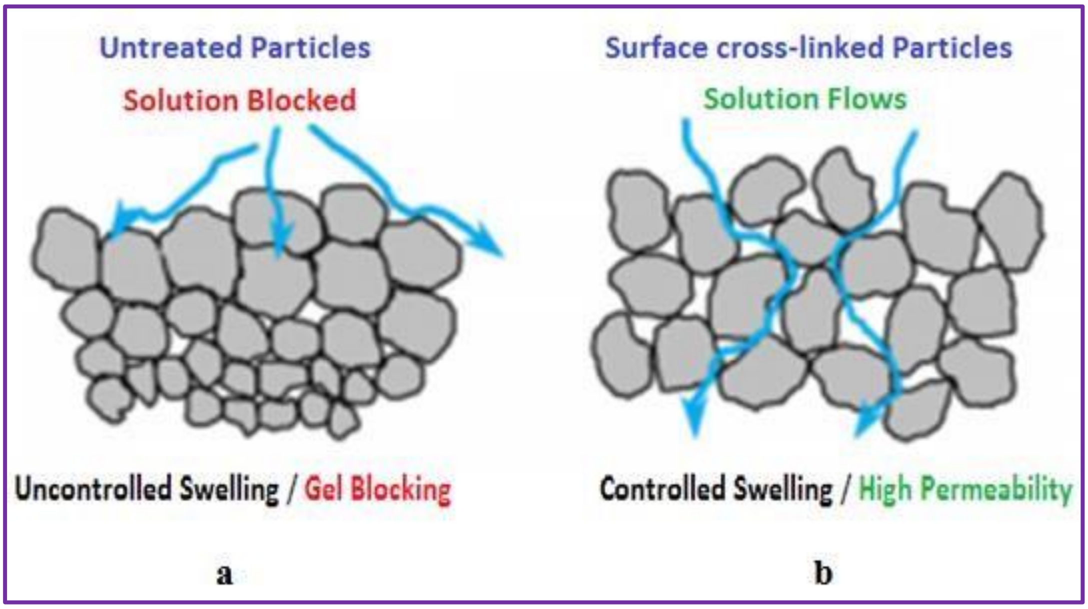

Figure 8: Effect of surface cross-linking on the solution flow [18]

In contrast, SAP with no surface treatment tends to show high swelling capacity but possess poor absorption versus pressure. Unfortunately, this way to improve the swelling capacity by reducing the cross-linking density, usually restricted by the increasing in extractable content of the hydrogel.

Furthermore, the pressure on these untreated particles can cause a "gel blocking" which prevents further liquid penetrating the gel bed. To obtain cross-linking density in the surface higher than in the bulk (Figure 9), particles subjected to the cross-linking solution, then 'cured' by heating [2].

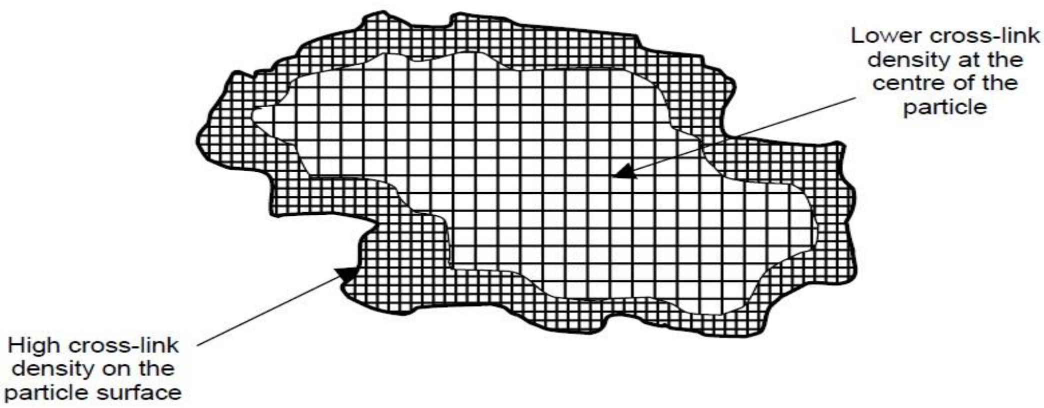

Figure 9: A surface cross-linked superabsorbent particle [2]

2-4 Super Absorbent Polymer Applications

Due to their outstanding characteristic, super absorbent polymers (SAPs) have been used in many applications. Figure 1 reflects some of these uses.

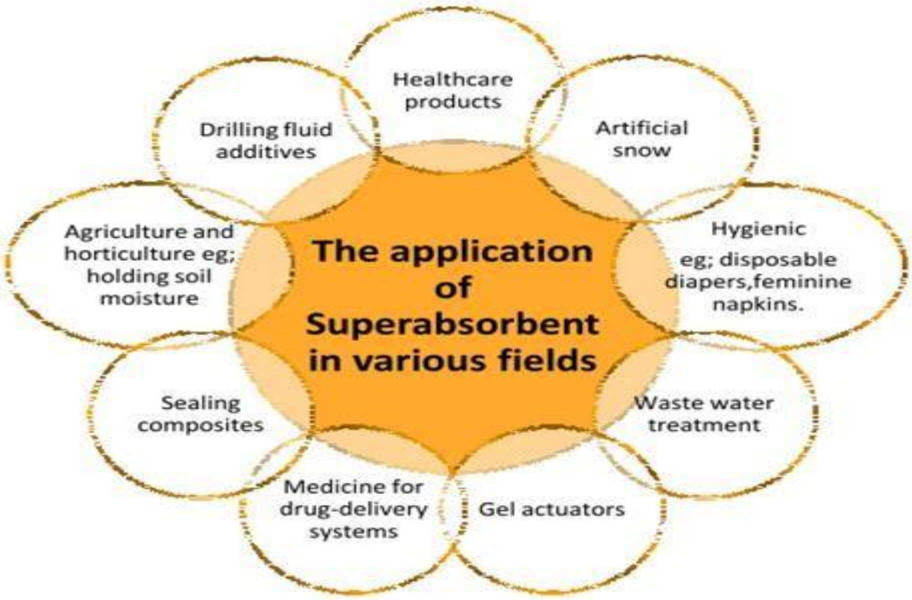

Figure 1: SAPs applications in various fields [19]

2-4-1. Smart Applications

Smart hydrogels are sensitive to environmental changes which may be physical or chemical variations like fluctuations in magnetic field, photoirradiation pH, temperature, urea, glucose and solution's ionic strength (Figure 2). Smart hydrogels used in many applications such as biosensors, actuators (artificial muscles), self-regulating drug delivery systems, on–off regulation of enzymatic reactions and purification of chemical agents .

The pH-response polymer gel is an example of smart hydrogel which can responds to the environmental pH variations similar to variations occur within the human body. pH values can change greatly between injured and infected tissues. For example normal tissue fluid has a pH ≈7.4, but it can be as low as 3.5 in a wound, and increase to 9.0 for an infected wound tissue [20].

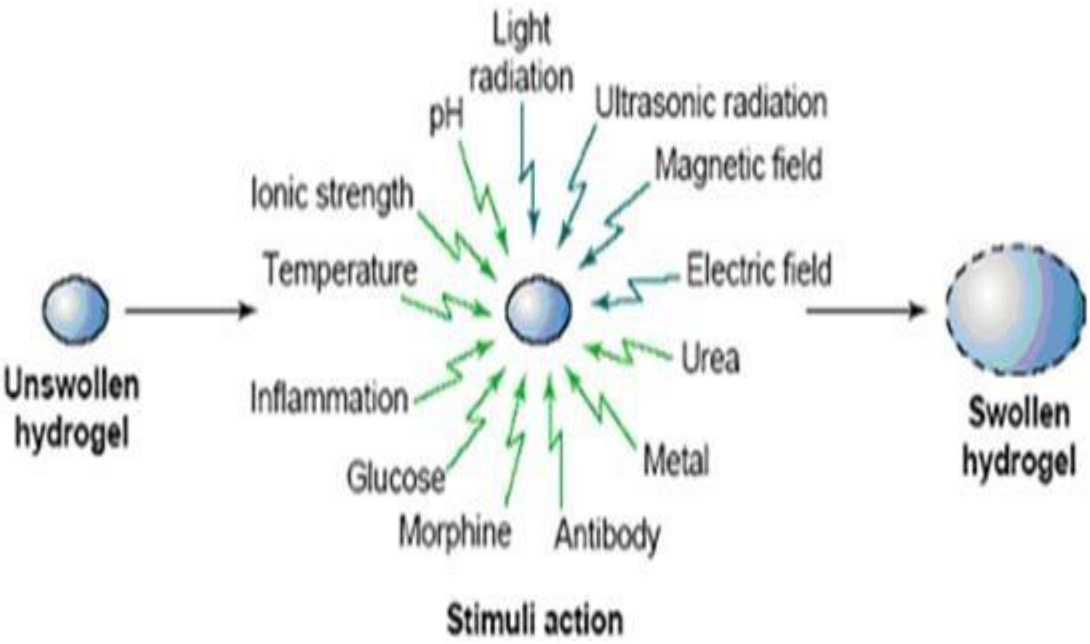

Figure 2: Swelling behaviors of intelligent hydrogels [19]

Normally, the pH- responsive gel possesses a molecular structure contains cross-linked network with ionize-able groups within its network. These groups will ionize in different ionization levels analogs to the pH and ionic strength levels.

As the network structure changed with the surrounding pH, the H- bond is converted also, which in turn created changes in mass and volume of the gel.

2-4-2. Medical Applications

SAPs can be used in many medical applications, such as drug delivery systems (DDSs), wound closure , healing products, removal of body water during surgery(treatment of edema), and surgical implant devices[.21]. SAPs, also used in the health care field to manufacture an efficient heating pads to help patients whom suffer from rheumatism.

One example of the control on–off drug release is the sugar responsive gel which is used for diabetes treatment. In healthy physiological conditions, cells in the pancreas releases insulin hormone to control blood glucose level within the range of 70 - 110 mg/dl. In contrast, for patients whom cannot control blood glucose levels, it is necessary to added insulin externally. Hypoglycemia and coma may result from the irregular doses, thus insulin must be accurately added.

Insulin hormone is produced from beta cells in the pancreas to control glucose levels in the blood. Diabetes disease arises when there is no enough production of this hormone [22].

Recently, super-porous hydrogels (SPHs) have been developed with very fast swelling behavior due to their open porous structure in order to controlling the delivery of drugs to intestine or stomach, as well as for gastric retention applications [23].

2-4-3. Agricultural Applications

SAPs inside the soil can absorb water from rainfall or excessive irrigation, then releasing it slowly upon the root demand through osmotic pressure difference, resulting in improved growth rate. Furthermore they can be used for the controlled release of pesticides and agrochemicals [24]. SAP hydrogels can influence many properties of soil such as permeability, aeration, density, structure, erosion resistance, evaporation, microbial activity and infiltration rates. SAPs also used to increase both the nutrient retention and the water-holding capacity for sandy soils (macro porous medium) in the arid areas [25]. Furthermore, SAPs can reduces both of the losses of water, irrigation frequency and soil compaction tendency [26].

For plants, SAPs can increase color intensity, coverage percentage and sport turf density, and decreases it's wilting level. SAPs in agriculture can be used as mixtures with soil, seeds, agricultural chemicals, and fertilizers(called water-absorbent slow release fertilizer). For forestry field, SAPs is covered over the roots to protect tree from drying during transportation

2-4-4. Strengthening of Concrete Application

Although mortar and concrete are popular construction materials, but they sever from some weaknesses such as late hardening, large drying shrinkage, low chemical resistance, and low tensile strength.

SAPs can act as an internal water source to provide hydration process with the required water according to the following mechanism [27]:

1- SAPs absorbs water molecules from the fresh concrete mixture, then releases it in a later stage if its relative humidity decreases because of the hydration process.

2- When SAPs have got their final sizes, they establish stable, water-filled inclusions, then water will sucked from these inclusions into smaller capillary holes and consumed by the cement hydration action [28]. SAPs can be used to promote internal curing of concrete; thus mitigate its autogenous shrinkage. This is because internal curing allows curing to be 'from inside to outside' by the created internal reservoirs; thus provides water throughout the mix not just penetration for a few millimeters such as in external curing (Figure 3) [29].

Also, the created gel can improve both the workability and stability due to provide the concrete mass with cushioning and lubrication effects [30].

Effective mortar properties are the consistency and the setting times.Consistency of standard paste refers to the water content of the paste which will produce the wanted consistency.

Setting indicates the change from a fluid state to a rigid state and mainly caused via the hydrationoftricalcium silicate(C_3S) andtricalcium aluminate (C_3A) and occurs in two stages; initial and final sets.

Setting is important in concrete work to keep fresh concrete plastic for enough time which helps the processes of transporting, casting, and compaction [29].

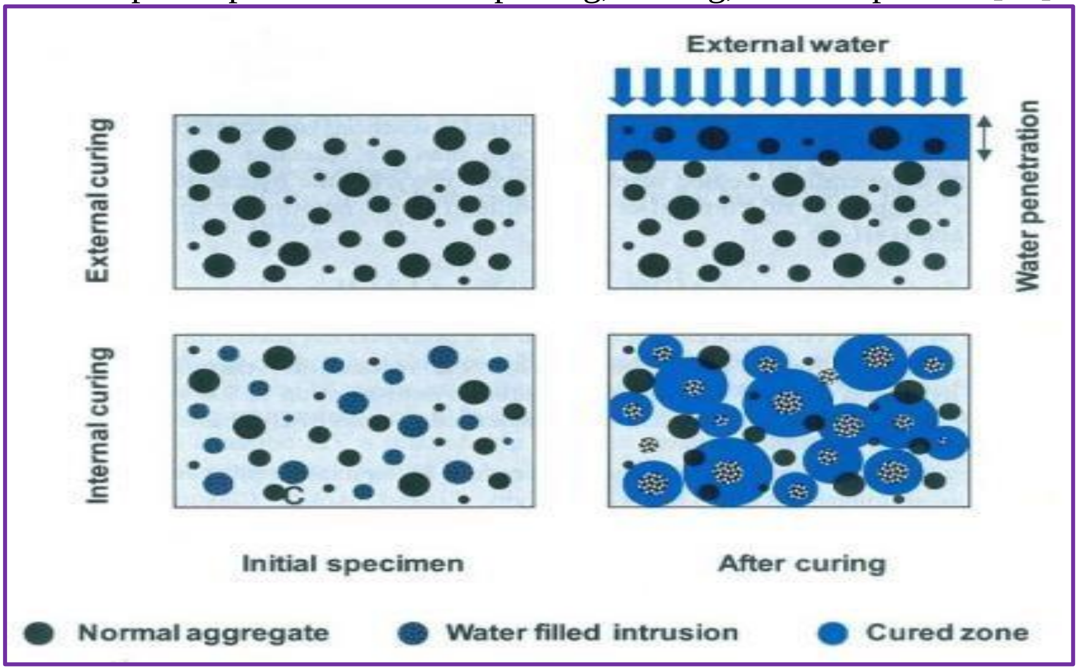

Figure 3: Internal curing vs. external curing [29]

Initial set considers as the time elapsed since the moment of mixing water with the cement to the moment of penetration of no deeper than 4-6 mm from the bottom. The minimum initial set time is45 minutes according to the ASTM C 150-09.

The final setting is usually calculated from the moment of mixing water with the cement. There are no limits appearing in the European or ASTM standards for the final setting time [30].

2-4-5. Other Applications

SAPs can be used also in many applications such as, fire retarders, dewatering of adulterated fuel, artificial snow [31], athletic garment, ornamental (colored) products, sanitary towels, building internal decoration[32], modifying weather condition, as dehydrating agent to solidify waste in the sludge treatment ,absorb alcohol as well as the heavy metal ions like Co^{2+} and Cr^{3+},extract urea from urine in artificial kidneys (urea absorbing material), to improve the moisturizing effects of different cosmetic products and cleaning radioactivity from porous structures [32].

CHAPTER THREE

EXPERIMENTAL PART

3-1Iintroduction

This experiment was conducted in agricultural applications, and two types of plants were used :

1- Cress seeds

2-Been seeds

Different quantities of super-water-absorbing polymers were placed in each of them, and the effect of each percentage on the plant was studied, with several tests being conducted, which we will discuss later.

3-2Materials

Cress seeds, bean seeds, and a superabsorbent polymer were used

3- 3 SAP characterization

3-3-1 **FTIR** test

The FTIR test was done using the international standard ASTM E1252

The infrared spectra that were obtained through ftir can help determine the chemical composition or the bonds in an unknown molecule. The sample is prepared and mixed with the (KBR) and fixed to the device and repeated rays on it and the sample absorbs and emits other and through this we can identify the identity of the material.

3-3-2 **DSC** test

The DSC test was carried out using the international standard ASTM D3418

Quality control by thermal analysis is used to inspect products for irregularities that could compromise their quality. For example, by checking the level of crystallinity and the magnitude of the glass transition of an injection-molded part, one can measure the effects of cooling within the mold
The most important effects that can be analyzed by DSC are the melting behavior, glass .transition, chemical reactions, and the effect of fillers

3-4 SAP morphology test

The SEM Examination Was Carried Out According To The International Standard ASTM E1508
A scanning electron microscope is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with the atoms in the sample, producing various signals that contain information about the topography and composition of the surface.

3-5 polymer extraction test

It was made using the international standard ASTM D2172

(1) g of the polymer was prepared and mixed with 20 ml of ethanol in a magnetic mixer for a full hour, then the polymer was filtered and dried in the electric oven. The weight of the
: polymer was measured again and the equation was applied

Extract=W1_W2/W1×100%............................(3,1)

3-6Water absorbance capacity and absorption kinetic test

It was made using the international standard ASTM D570 Five samples were prepared, each one containing (1) g of polymer, immersed in distilled water and left for an hour to measure the absorbance according to the equation mentioned and (15-30-45-60-90) minutes, respectively, to measure the adsorption kinetics, and then : dry the polymer and measure its weight again and apply the mentioned equation Se=W1-W2/W1…………………..(3,2)

3-7 Acidity degree (pH) test

It was made using the international standard ASTMD1293. pH examination and this examination was carried out according to international standards.

The pH value is used as a standard scale for measuring the properties of water

In scientific terms, the pH value expresses the concentration of hydrogen ions (H+) in a liquid. If the concentration of hydrogen ions (H+) in the liquid is high, it is acidic. If the concentration is low, it is basic (alkaline). The pH value has a scale from 0 to 14, where 7 is the neutral level and any value less than 7 increases the level Acidity with decreasing value, and any value higher than 7 is alkaline and alkalinity increases with increasing value

3-8 Water retention test

It was made using the international standard ASTM C1506

1g of the polymer was taken and placed in 100ml of water for an hour, then left to dry for ten minutes and weighed again. The amount of water retained inside the polymer can be

: calculated from the equation

Water retention=W2-W2/W1………………. (3,3)

3-9 Ionic sensitivity test

(1)g water for a full hour, then dried, filtered and measured for its weight and absorbency.

The same amount and age were taken in 100ml of water containing 0.2 of salt and its weight was measured again and its absorbance calculated, after which the ionic sensitivity : can be calculated from the following equation

F=1-Se in given fluid/Se in DW…………………(3-4)

3-10Absorption kinetics for saline solution

It was made using the international standard ASTM D570 Five samples, all containing 1g of polymer, were prepared and placed in 100ml of water containing 0.2% of salt for an hour to measure the absorbance. These samples were withdrawn from the water during (13-30-45-60-90) minutes, respectively, to calculate the kinetics of absorption. They were dried and weighed. New and calculate the absorbance

: from the equation

Se=W2-W1/W1………………. (3,4)

3.11 Agricultural uses of the current SAP

3.11.1 Cress seeds

It is a type of vegetable that is widely cultivated in Iraq, and its cultivation requires clay soil in order to retain water, because cress needs constant water. As for the time of its cultivation , it is annual plants with cold weather.,They are grown in the garden,early spring,Until 4 or 6 weeks before the last frost. Cress is also grown indoors all year round,It is grown in late summer and winter,It is a winter herb that can be grown from mid-August to late December. cultivation method:The soil to be planted was prepared and divided into three equal plates, each 50 cm long and 25 cm wide. We put 5 g in the first plate, 10 g in the second plate, and in the third plate we did not put any amount of polymer . Cress seeds were planted in it and then watered daily, and after germination of these seeds, it was watered once every four days

3.11.2 Green Beans

The broad bean is one of the main legume crops. The bean plant needs confined temperatures between6-30 ° C, as it is sensitive in low temperatures less than 4 ° C, especially during the flowering and seed formation stage.,But if the degree exceeds 30, it leads to the fall of flowers from the plants, and the legumes are considered one of the plants most present in humid areas, and they need an important amount of moisture at the soil level, especially in the first periods of growth. It is preferable to plant legumes at the beginning of October cultivation method:Five special plastic bags were prepared for growing plantsWe put an equal amount of soil (1000g) in each bag and put an amount of polymer in each bag at a distance of 2 cm from the surface of the soil and leveled it.

We put in the first bag 0.005g of the polymer and in the second bag 0.01g

And in the third bag 0.015 g and in the fourth bag 0.02g and we left the fifth bag without any polymer, and we planted beans in each bag and watered them daily, and after germination they were watered once every four days

CHAPTER FOUR

Results & discussions

-

4.1 Introduction

This chapter contains with the sections:

1. Characterization of the used SAP using different techniques, such as FTIR and DSC. The used SAP was imported from CHINA under a trade name of SAP-92 without specifying its chemical structure.
2. Study the morphology of this polymer to investigates its ability to absorb water during plant watering action. Thus, SEM technique used.
3. The used SAP was evaluated in terms of polymer extraction and water retention.
4. Many related SAP properties were measured, such as ionic sensitivity and acidity degree.
5. The absorbance capacities and its kinetics were calculated with ordinary water and with saline solution containing 0.9% NaCl to mimic the absorbance in swampy lands (Salt marshes).
6. study the effects of SAP in agricultural application on the growth of two plants; Cress plant and Green Beans. These effects monitored in terms of length, weight, color intensity, density, flowers number and so on.

4.2 SAP characterization

4.2.1 FTIR results

Figure 4.1 shows the FTIR spectrum of the used SAP. Band around 3600 cm-1 belongs to N-H or O-H groups. Bans from 2800 to 2900 cm^{-1} are due to the stretching and bending of C-H group. Band at 1620.51 cm^{-1} is due to the carbonyl group (C=O). This means that the used SAP is from Acrylic polymeric family, which is often used absorbing water applications.

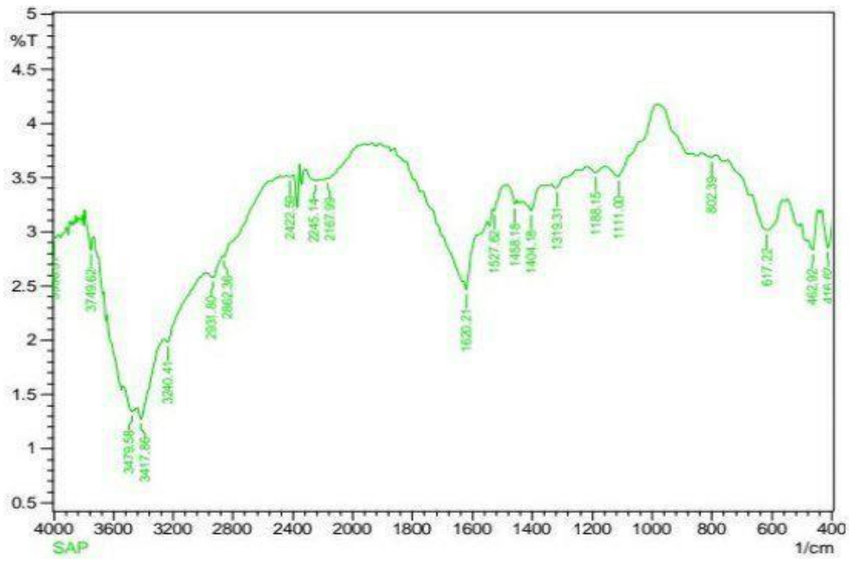

Figure 4.1: FTIR spectrum of the used SAP

4-2-2 DSC results

According to the DSC curve (Figure 4.2), the used SAP have high thermal stability and its structure does not affect during hot environments. Its glass transition temperature Tg is about 80°C and its melting temperature Tm is more than 250, which indicates the high mechanical properties, which qualifies it for use under harsh conditions, such as those it is exposed to when mixed with soil near the roots of plants, under varying thermal conditions.

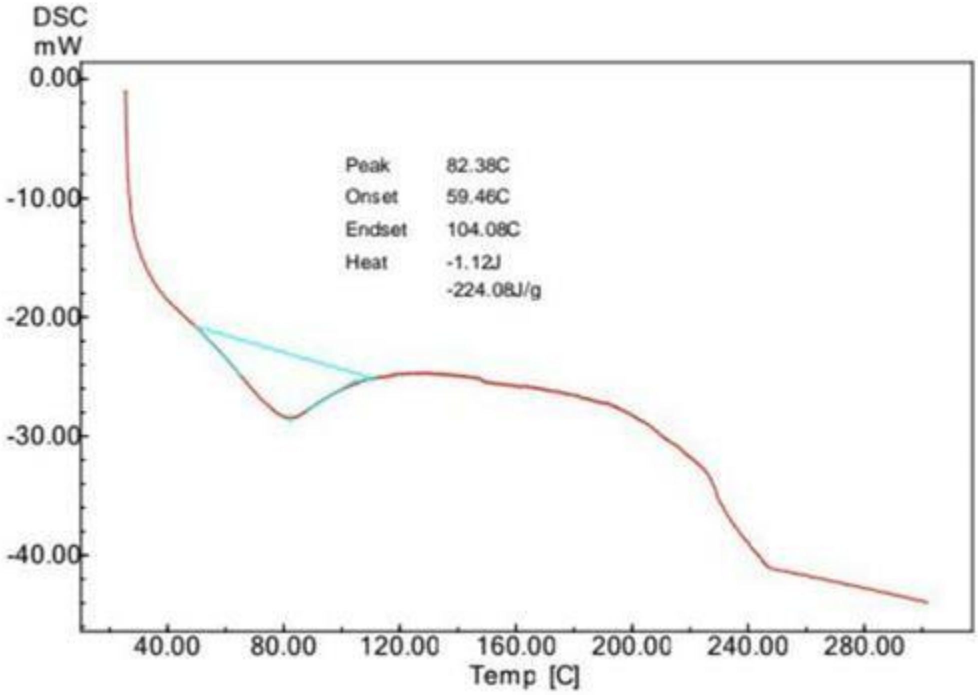

Figure 4.2: DSC curve of the used SAP

4-3 SAP morphology

It is clear from figure 4.3, that the used SAP have rough surface containing many pores, grooves and cavities and pores, which enhancing water absorbing and maintaining water inside these cavities and provide plant with the required quantity of water during summer season. This surface nature encourages the used SAP to be hydrophilic tendency.

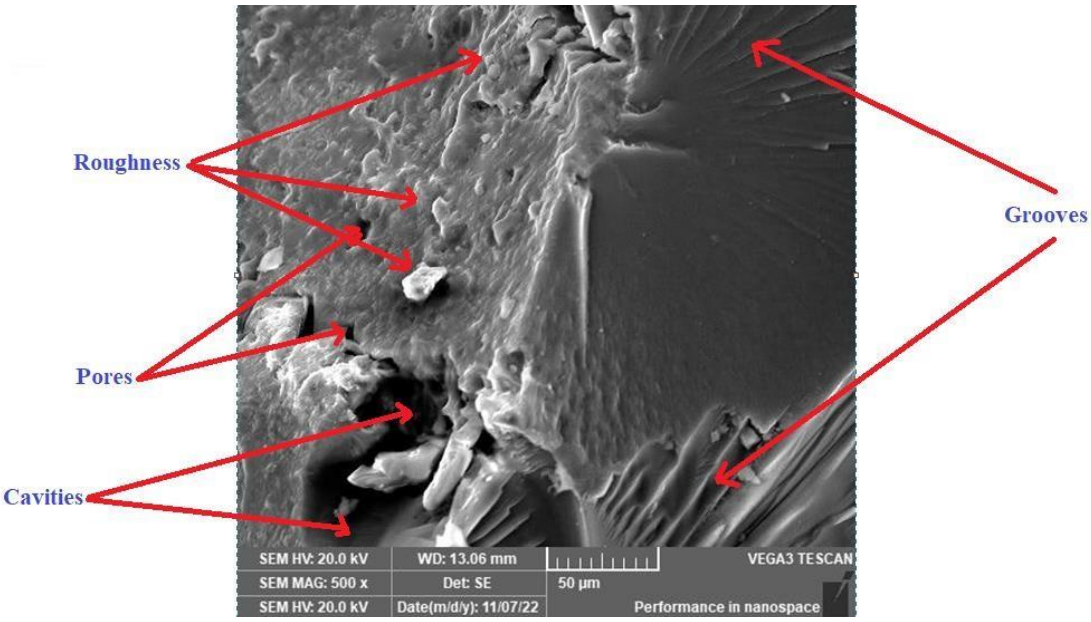

Figure 4.3: SEM image of the used SAP

4-4 Polymer extraction results

This test done to evaluate the safety of the used SAP during swelling with water and other dissolving media and its dissolution resistance. The test also gives an indication about the cross-linking density among SAP chains. The lower the result of the test, the more the SAP retains its physical structure.

Results showed that the polymer extraction ratio is 29%, which means that:

1. The used SAP losses about 29% of its initial weight during the swelling action.
2. There is a lot of unreacted monomers; the polymerization process was uncompleted.
3. There is a lot of chain ends, which means that this polymer have high molecular weight distribution.

That means that there is a real need to improve the chemical structure of this polymer to reduce its weight losses by increasing the cross-liking density, for example.

4-5 Water absorbance capacity and absorption kinetic

The absorbance capacity of the used SAP was measured according to eq. 3. And its found to be 34.09 g/g after 90 min. This means that the used SAP have sufficient ability to absorb water. This good ability is due to the chemical functional group, such as the hydroxyl group (OH) and N-H group, which can form hydrogen bonds with water. Also, the rough surface causes the SAP to kept water within the SAP structure.

The absorption kinetic was studied from 15 to 90 minutes with 15 minute as interval time as shown in table 4.1 and figure 4.4.

Table 4.1: Water absorbance kinetic for the used SAP

Time (min)	Absorbance (g/g)
15	21.47
30	26.1
45	29.33
60	26.08
90	34.09

It is clear that, with the time the absorbance increased linearly (to some extent) from 21.47 to 34.09 g/g. This means that water molecules continue in diffusing among the polymeric chains; making these chains to be apart by increasing the distance between these chains. Absorbance kinetics, also, showed that the SAP does not reach to the equilibrium swelling up to 90 minute and need an extra time to stop the swelling tendency. This means that the swelling is gradual and not happened suddenly.

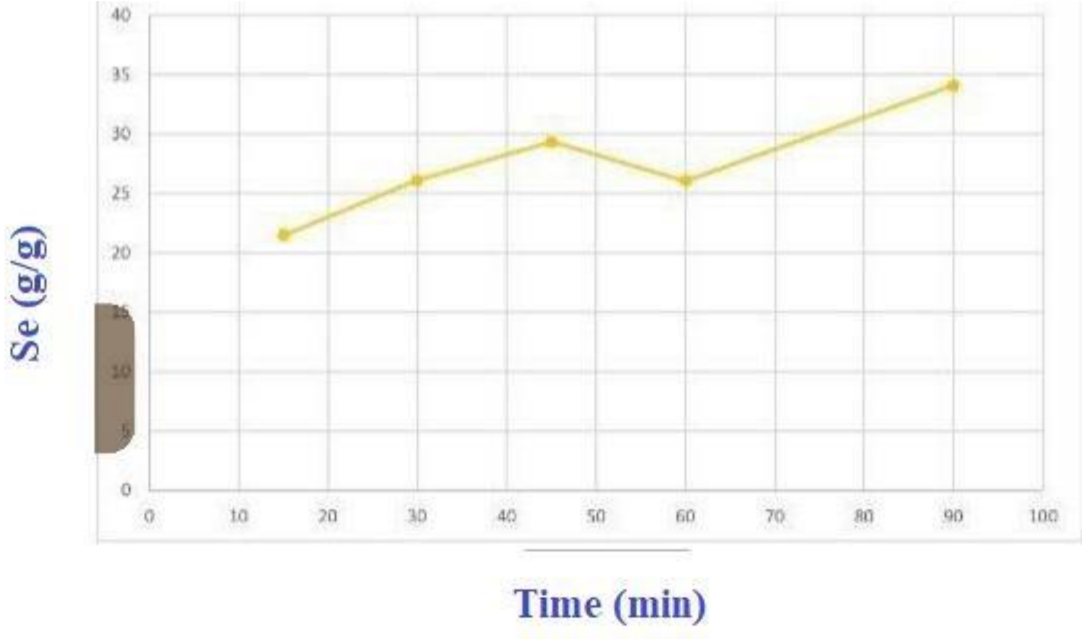

Figure 4.4: Water absorbance profile for the used SAP

4-6 Acidity degree (pH)

The acidity degree of the used SAP was measured using pH-meter and it is found equal to 7, which means that the used SAP is neutral and can be used in both acidic and alkaline environments.

4.7 Water retention results

The ability of the used SAP to maintain the absorbed amounts of water were monitored using this test (according to the equation 3)

, which shows that this SAP have poor retention ability; 33.03%. This bad result coincide with the low extraction result (29 %) . These two bad results indicate to that the used SAP dissolves with time and loss its structure due to poor cross-linking density. Therefore, this polymer needs chemical modification to increase its resistance to dissolution.

4.8 Ionic sensitivity results

The sensitivity of the used SAP was measured using the dimensionless welling factor, f, which is defined as follows:

f = 1- (Absorption in a given fluid / Absorption in distilled water) For current SAP, the calculated f is 2.52, which means that there is a higher absorbency-loss of the sample swollen in salt solutions. Therefore, SAPs with lower f are usually preferred. Negative values of f reveal that the absorbency is not decreased, but, it is increased in salt solutions. The SAP hydrogels with betaine structures exhibit such surprising behavior.

4.9 Absorption kinetics for saline solution results

To mimic the absorbance behavior of swampy lands in agricultural application, the absorption kinetic in saline solution (0.9% NaCl) was studied from 15 to 90 minutes with 15 minute as interval time as shown in table 4.2 and figure 4.5.

Table 4.2: Saline absorbance kinetic for the used SAP

Time (min)	Absorbance (g/g)
15	20.626
30	21.969
45	23.206
60	26.639
90	30.683

It is clear that the absorbance behavior in 0.9% NaCl solution is similar behavior to that in distilled water, but with less absorbance capacities within the saline solution. That means the possibility of using SAP with swampy lands)Salt marshes(. Also, this polymer does not reach to the equilibrium absorbance within 90 minute and need more time.

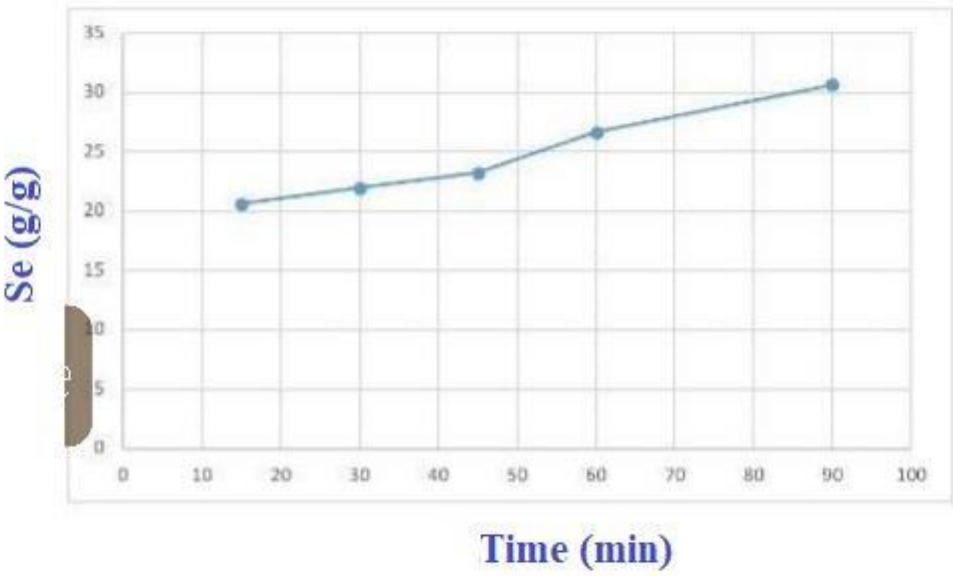

Figure 4.5: Saline absorbance profile for the used SAP

4.10 Agricultural uses of the current SAP results

In current work, the SAP was used in two different ways. The first application is ground planks for cress plants, where the SAP was mixed with the soil of the plank in different proportions (0,5 and 10 wt%), and the growth of the cress plant was compared with a reference plank free of SAP.

In the second application, SAP was mixed with the soil incubating the bean seeds in several proportions in agricultural stocks, and it was compared with soil free of the polymer.

The presence of water in soil is essential to vegetation. Liquid water ensures the feeding of plants with nutritive elements, which makes it possible for the plants to obtain a better growth rate. It seems to be interesting to exploit the existing water potential by reducing the losses of water and also ensuring better living conditions for vegetation. Taking into account the water imbibing characteristics of SAP materials, the possibilities of its application in the agricultural field has increasingly been investigated to alleviate certain agricultural problems.

SAPs have been successfully used as soil amendments in the horticulture industry to improve the physical properties of soil in view of increasing their water-holding capacity and/or nutrient retention of sandy soils to be comparable to silty clay or loam.

SAP hydrogels potentially influence soil permeability, density, structure, texture, evaporation, and infiltration rates of water through the soils. Particularly, the hydrogels reduce irrigation frequency and compaction tendency, stop erosion and water run off, and increase the soil aeration and microbial activity. In arid areas, the use of SAP in the sandy soil (macroporous medium), to increase its water-holding capacity seems to be one of the most significant means to improve the quality of plants.

The SAP particles may be taken as "miniature water reservoirs" in soil. Water will be removed from these reservoirs upon the root demand through osmotic pressure difference.

The hydrogels also act as a controlled release system by favouring the uptake of some nutrient elements, holding them tightly, and delaying their dissolution. Consequently, the plant can still access some of the fertilizers, resulting in improved growth and performance rates.

On the other hand, SAPs in agriculture can be used as retaining materials in the form of seed additives (to aid in germination and seedling establishment), seed coatings, root dips, and for immobilizing plant growth regulator or protecting agents for controlled release.

4.10.1Results of Cress seeds

Figure 4.6 shows images of growth of cress plant with different SAP ratios (0, 5 and 10 wt%) after 6 and 16 week. It is clear that the 5 wt% SAP addition increased the Cress vegetation. Also, the leaves color of the plant has become brighter and fresher. On the other hand 10 wt% addition caused the death of some plants and reduced the color intensity of the leaves. Therefore, the 5 wt% represents the best ratio for obtaining the desired vegetation.

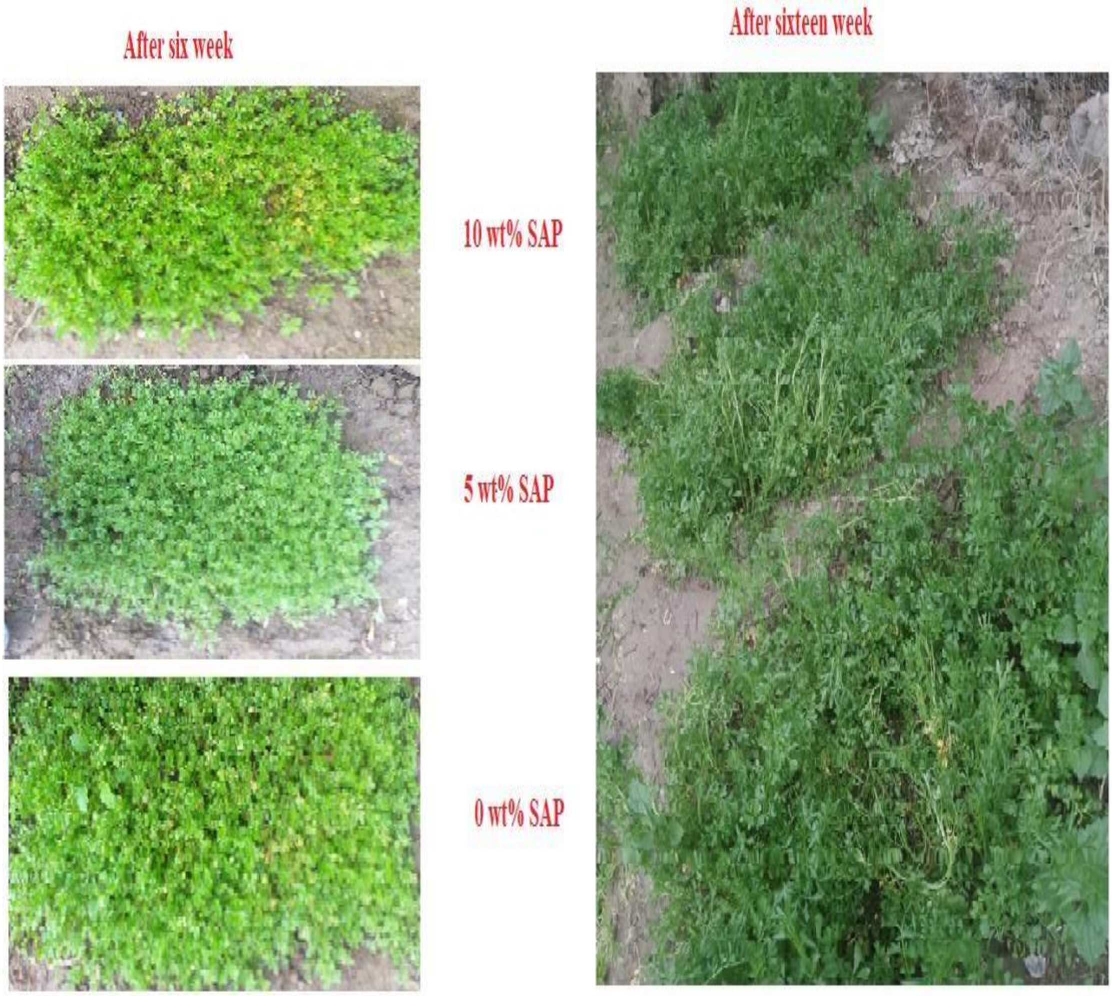

Fig. 4.6: Images of cress plant with different SAP ratios after 6 and 16 week

After 16 week, the length of the Cress plant also measured (Figure 4.7). It is clear that the plant length increased linearly with the SAP content from 41 cm to 47 cm and up to 57 cm. But the 5 wt% SAP gives maximum number of branches of the plant ; better vegetation and better density. Therefore, the 5 wt% represents the better one.

That means SAPs inside the soil absorb water from rainfall or excessive irrigation, then releasing it slowly upon the root demand through osmotic pressure difference, resulting in improved growth rate. Furthermore they can be used for the controlled release of pesticides and agrochemicals. It is expected that SAP hydrogels can influence many properties of soil such as permeability, aeration, density, structure, erosion resistance, evaporation, microbial activity and infiltration rates. SAPs also used to increase both the nutrient retention and the water-holding capacity for sandy soils (macro porous medium) in the arid areas. Furthermore, SAPs can reduces both of the losses of water, irrigation frequency and soil compaction tendency.

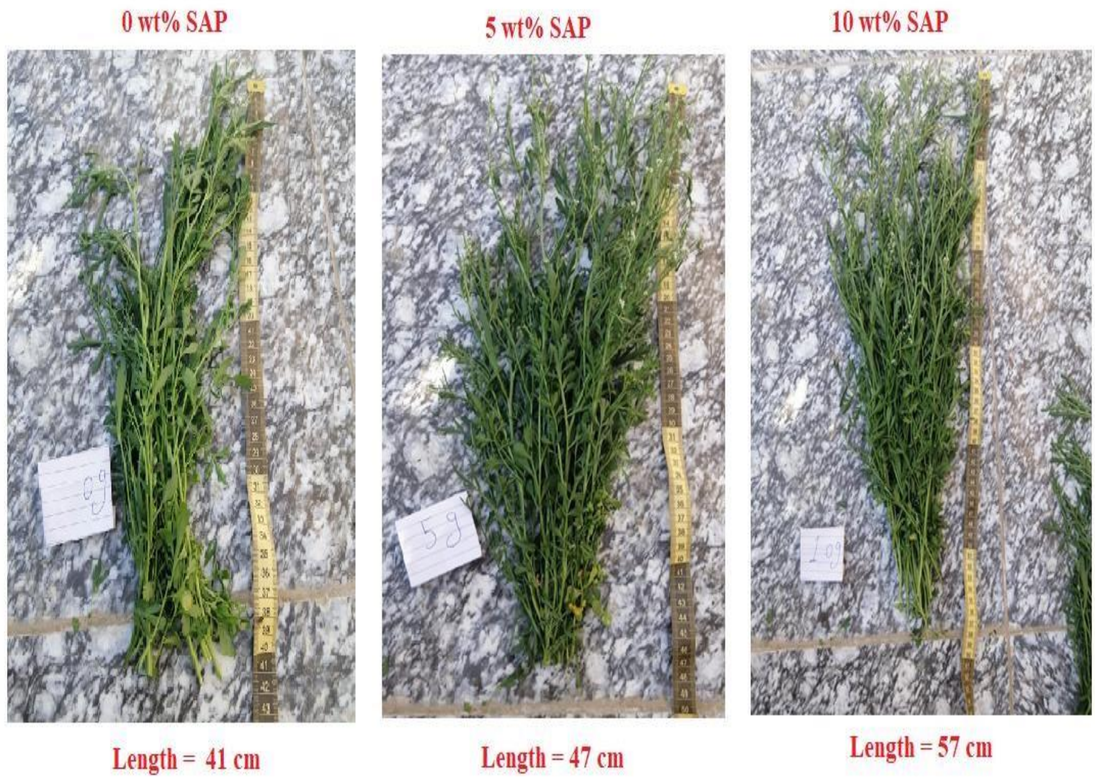

Fig. 4.7: Lengths of cress plant with different SAP ratios after 16 week

Also, the weights of the Cress plant measured after 16 week, as shown in table 4.3. It is clear that the weight increased as SAP content increased. Table 4.3: Weights of cress plant with different SAP ratios after 16 week .

SAP (wt%)	Weight (g)
0g	23.9
5g	39.1
10g	62.8

4.10.2 Results of Green Beans

The effects of SAP upon the growth of Green Beans where monitored by images after 8 weeks. It is clear that SAP have a positive effect of on the growth ability. For plants, SAPs can increase color intensity, coverage percentage and sport turf density, and decreases it's wilting level. SAPs in agriculture can be used as mixtures with soil, seeds, agricultural chemicals, and fertilizers (called water-absorbent slow release fertilizer). For forestry field, SAPs is covered over the roots to protect tree from drying during transportation

Fig. 4.8: Growth of Green Beans with different SAP ratios after 8 week

Also, the number of flowers were calculated after twenty weeks for different SAP ratios (0,5,10,15 and 20 wt%) as shown in table 4.4. It is clear that number of flowers increased from 9 to 15 and then up to 28 flower at 10 wt% SAP. After that, the flowers decreased to 20 and 13 flower at higher SAP ratios. This means that the 10 wt% ratio is the best ratio.

Table 4.4: Number of flowers for Green Beans with different SAP ratios after 20 week

SAP (wt%)	number of flowers
0	9
5	15
10	28
15	20
20	13

CHAPTER FIVE

CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions

1. The used SAP belongs to Acrylic family and its high absorbance capacity is due to its active chemical functional group as well as to its surface roughness.

2. The used SAP have sufficient mechanical and thermal properties, but suffer from the presence of unreacted monomers (losses about 29% of its initial weight during swelling) and high amounts of end chains. This shortcoming investigated also in the ionic sensitivity test, which give high positive value; f = 2.52.

3. Also, this polymer suffers from low retention ability (only 33.03%), which means the need to modify the chemistry of this polymer.

4. The used SAP shows good absorbance capacities with both ordinary water and saline solution (0.9% NaCl), which refers to possibility of used it in various lands types. After 90 min the water absorbance is 34.09 g/g and 30.683 g/g for saline solution.

5. The absorbance kinetics with ordinary water and saline solution shows the gradual absorbance with time but do not reach the steady-state before 90 minute.

6. The used SAP is neutral and can be used in neutral, acidic and alkaline environments.

7. Results of Cress plant showed that the 5 wt% represents the best ratio for obtaining the desired vegetation, while with 10 wt% some plants died and the color intensity of the leaves reduced. Weight and length of the Cress plant increased linearly with the SAP content but the 5 wt% SAP gives maximum number of branches of the plant, better vegetation and better density.

8. Results of Green Beans showed that a positive effects of on the growth ability, color intensity, coverage percentage and flowers number.

5.2 Recommendations

1. Use the current SAP in another agricultural applications, such as stabilizing the sport turf, green belts and combating desertification.

2. Loading current SAP with fertilizer and study its release.

3. Study another plant properties, such as the weight of dried plants and the

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