

# Enhancing Irrigation Efficiency Through The Integration Of Potassium-Based Hydrogel In Ldpe Mulch Film For Sustainable Agriculture

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## Abstract

Irrigation plays an important role on our daily basis. All consumable green plants are being irrigated under manual supervision. A proper level of water and sunlight (humid condition) is the basic necessity for proper growth and health of the crops. Mulching is the process being followed in places with extreme climate conditions where scarcity of water is high. On the other hand, on recent growth the hydrogels are being used on large scale for agriculture purpose for their tendency to absorb and preserve water.

Thus, using both the concepts, the hydrogels are being incorporated in the mulching film to increase their usage in the field of plasticulture. Mulching film is prepared using Low Density Polyethylene (LDPE) by extrusion process. Potassium based hydrogels are used to improve the production and to maintain the pH of the soil. Thus, mulching film incorporated with potassium-based hydrogels can improvise the production of crops and minimize the usage of surplus water maintaining the nutrition of the soil.

**Keywords:** Potassium based Hydrogel; Development in irrigation; Improvisation of mulch film; Polymers in agriculture; LDPE with hydrogel; Advancement of polymer in agriculture.

## INTRODUCTION :

### Objective of the work

The mulching technique has been followed in the arid climatic conditions where water availability is scarce and evaporation is to be avoided. Also, hydrogels are recently used directly on the field which helps to absorb water and deliver it to the crop when required. Thus, instead of using two different techniques, the project focuses on interlocking hydrogels in the mulch film so that a single process can be done. The hydrogel incorporated mulch film helps in conserving evaporation as well hydrogels help to conserve water usage.

### 1. Introduction

Low density polyethylene is the majorly used commodity plastic used by all common people. It is mainly preferred for its easy availability and low cost.

#### 1.2.1 Mulching Process

Mulch is a layer of material applied to the surface of [soil](#). It is a layer of organic or inorganic material placed over the root zone of a plant to benefit the roots and the soil. Organic materials may include wood chips, bark, pine needles, and straw, leaves, or grass clippings. These materials will eventually decompose, adding organic matter to the soil and need to be supplemented or replaced on a regular basis. Inorganic materials typically do not

decompose, need replacement only occasionally, are often more expensive, and include rock or pebbles, landscape fabric, newspaper, plastic or even shredded rubber tires for playground areas.

Mulching is the process or practice of covering the soil/ground to make more favourable conditions for plant growth, development and efficient crop production. Mulch technical term means 'covering of soil'. While natural mulches such as leaf, straw, dead leaves and compost have been used for centuries, during the last 60 years the advent of synthetic materials has altered the methods and benefits of mulching. The research as well as field data available on effect of synthetic mulches make a vast volume of useful literature. When compared to other mulches plastic mulches are completely impermeable to water; it therefore prevents direct evaporation of moisture from the soil and thus limits the water losses and soil erosion over the surface. In this manner it plays a positive role in water conservation. The suppression of evaporation also has a supplementary effect; it prevents the rise of water containing salt, which is important in countries with high salt content water resources.



**Fig 1. Mulching film**

Advantages on using plastic mulch

- To suppress weed growth

Sunlight helps foster the growth of weeds by compacting soil; plastic mulch blocks sunlight from reaching the soil around the plant, thus preventing weeds from appearing. Black polyethylene and other opaque films in particular are useful for this purpose, blocking a wider range of weeds than organic mulches. The only exception is clear plastic, which lets sunlight in. The presence of fewer weeds also reduces the necessity for mechanical cultivation, leading to less root damage.

- To conserve water in crop production and landscaping

Plastic film with its moisture barrier properties does not allow the soil moisture to escape Water

that evaporates from the soil surface under mulch film, condenses on the lower surface of the film and falls back as droplets. Thus moisture is preserved for several days and increases the period between two irrigations. The irrigation water or rainfall either moves into the soil through holes on the mulch around the plant area or through the un-mulched area.

- Better moisture retention

#### Mulching

facilitates even distribution of water and hence soil can retain water for a longer time. It ensures each single crop gets enough water for its well growth.

- Minimized fertilizer leaching

#### Plastic mulches

prevent an excess of water from going into the soil, helping to limit the loss of plant nutrients through leaching. Because the mulch is restrictive in this sense, it is most ideal to use it in conjunction with the drip irrigation method, which allows the addition of water and fertilizer in small amounts.

- Higher crop quality

#### Plastic mulch

helps crops avoid direct contact with soil; this results in the plants being cleaner, which is desirable for fruits such as strawberries. It also works to prevent rot as mud from irrigation is not splashed on the crops.

- Reduction in root damage

The use of plastic mulch creates a practically weed free area around the plant, removing the need for cultivation except between the rows of plastic. Root damage associated with cultivation is therefore eliminated. Due to these factors, the use of plastic mulch can lead to an improvement in the overall growth of the plant.

According to the survey, there are different types of mulch film:

A wide range of plastic films based on different types of polymers have all been evaluated for mulching at various periods in the 1960s. LDPE, HDPE and flexible PVC have all been used and although there were some technical performance differences between them, they were of minor nature. Owing to its greater permeability to long wave radiation which can increase the temperature around plants during the night times, polyethylene is preferred. Today the vast majority of plastic mulch is based on LLDPE because it is more economic in use.

### Basic properties of mulch film

- a. Air proof so as not to permit any moisture vapour to escape.
- b. Thermal proof for preservation of temperature and prevention of evaporation
- c. Durable at least for one crop season

### Importance of parameters of the plastic film

#### (a) Thickness

Normally the thickness of the film does not affect the mulching effect except when it is used for solarisation. But some of the recent references do indicate the impact of film thickness on crop yield. Since it is sold by weight it is advantageous to use as thin a film as possible

but at the same time due consideration should be given for the longevity of the film. The early mulch film used was of 60-75 micron (240-300 gauges) thickness, and today it is possible to have 15 micron thick film due to advent of film extrusion technology. These films are mechanically weak, as shown by their easy tearing when pulled tension.

#### **b) Width**

This depends upon the inter row spacing. Normally a one to one and half meter width film can be easily adopted to different conditions.

#### **c) Perforations**

The perforations may be advantageous under some situations and disadvantageous for some other situation. The capillary movement of water and fertilizer distribution will be better and more uniform under unperforated condition. But for prevention of water stagnation around the plants, perforation is better. But it has got the disadvantages of encouraging weed growth.

#### **d) Mulch colour**

The colour of the mulch affects

- i. Soil temperature
- ii. Temperature of air around the plants
- iii. Soil salinity

**a.** Due to lesser quantity of water used

**b.** Due to reduction in evaporation and prevention of upward movement of water.

Transparent film - Deposits more salt on soil surface

Black film - Restricts water movement and upward movement of salt is reduced.

iv. Weed flora - Black film

v. Insect control - Opaque while film acts as golden colour and attracts insects

#### **Mulch Laying Techniques**

- i. Mulch should be laid on a non-windy condition
- ii. The mulch material should be held tight without any crease and laid on the bed
- iii. The borders (10 cm) should be anchored inside the soil in about 7-10 cm deep in small furrows at an angle of 45°.

#### **Pre planting mulch:**

The mulch material should be punctured at the required distances as per crop spacing and laid on the bed. The seeds/seedlings should be sown/transplanted in the holes.

#### **Precautions for Mulch Laying**

• Do not stretch the film very tightly. It should be loose enough to overcome the expansion and shrinkage conditions caused by temperature and the impacts of cultural operation.

• The slackness for black film should be more as the expansion, shrinkage phenomenon is maximum in this color.

• The film should not be laid on the hottest time of the day, when the film will be in expanded condition.

#### **Removal of mulch**

In case the mulch film needs to be used for more than one season (thicker film) the plant is cut at its base near the film and the film is removed and used. By compounding appropriate additives into the plastics it is possible to produce a film, which, after exposure to light (solar radiation) will start to breakup at a pre determined time and eventually disintegrated into very small friable fragments. The time period can be 60, 90, 120 or 150 days and for maize a 60-day photodegradable mulch is used. However there are still some further problems to resolve. It has been observed that the edges of the mulch, which are buried to secure the mulch to the soil, remain intact and become a litter problem when brought to the surface during the post-harvest ploughing.

Thus to overcome these problems, the mulch sheet is being replaced after every harvest. On the other hand, bio-degradable mulch sheet are also in use. But the major problem is that, the broken parts of the film lead to soil pollution. Thus these are the challenges that are to be overcome.

## 2. Hydrogel in Agriculture

Hydrogels are hydrophilic cross-linked polymers that form three dimensional molecular networks which can absorb and hold great amounts of water. Hydrogel agriculture technology uses insoluble gel forming polymers to improve the water-holding properties of different soils. This can increase water-holding and water use, improve soil permeability, reduce the need for irrigation, reduce compaction, soil erosion, and leaching, and improve plant growth.

Hydrogel Agriculture (Super Absorbent Polymer for Agriculture) is potassium based nontoxic polymer capable of absorbing and retaining water up to 300 — 500 times of its own size. When mixed with soil and sown at the roots of a plant, it spares 65 – 95% of water utilized. Its application is basic yet effective. It was created to develop farming in extreme climates, whether excessively hot, excessively parched, and so forth. It assists with: dry season, desertification, poor soil quality, and treatment. Rather than day by day watering, Super Absorbent Polymers permit watering once per week, sparing time, money, and water.

Super absorbent polymer for Agriculture can assimilate and hold a great degree of water in respect to their own particular mass. Water-retaining polymers, which are delegated hydrogels when cross-connected, absorb fluids through hydrogen bonding with water. A SAP's capacity to retain water is an element of the ionic concentration of the watery arrangement.

Super Absorbent Polymer(SAP) for Agriculture is effective for water utilization in agricultural and horticultural crops in areas with little or negligible amount of rainfall and under

limited irrigation conditions. It offers huge economic viability to cultivations and is a boon to dry states and the future generation of farming.

Hydrogel polymers/ Super Absorbent polymers (SAP) are a system of polymer chains hydrophilic in nature with water as the dispersion medium. Hydrogel polymer is capable of storing more than 90% water and has a level of adaptability same as natural tissues, because of their huge water content.

Hydrogel polymer is a water absorption agent that once added to soil or substrate will absorb and hold vast amounts of water and supplements, nearly up to 400 times of its own particular weight. Hydrogel polymers formed through cross linking polymer chains (physical, ionic or shared bonding) and are well known for their ability to absorb water. Hydrogels are mostly homogeneous in nature. Super Absorbent Polymers, which are delegated hydrogels, retain watery arrangements through hydrogen bonding with water atoms. In de-ionized and de-mineralized water, a SAP may assimilate 300 times its weight (from 30 to 60 times its own volume).

## Potassium based Super Absorbent Polymer (SAP)

Potassium Polyacrylate is a polymer of a potassium cation and acrylamide with an Absorption Capacity Index in the scope of 30-100. This polyacrylate helps in the germination of seedlings, with plants or for transportation of plants or seedlings. It is nontoxic, harmless, and non-polluting.

$$\text{Absorption Capacity Index (ACI)} = \frac{\left( \begin{array}{c} \text{Weight of water saturated} \\ \text{gel polymer} \end{array} \right) - \left( \begin{array}{c} \text{Dry weight of} \\ \text{polymer} \end{array} \right)}{\text{Polymer dry weight}}$$

Amid dry periods, sodium polyacrylate has a tendency to gather and form cross links that repress re-swelling when it is re-wetted. With a limited number of wet/dry cycles, sodium polyacrylate hinders plant development. This inhibition emerges because sodium particles in the sodium polyacrylate system are easily replaceable and these particles are absorbed by the soil particles or have a tendency of exchange with the cations on the surface of plant roots. The result is a condition that is different from an alkaline soil, which tends to affect and inhibit plant growth and soil hardening.

Hydrogels can be used in all type of agriculture such as

#### ***Open Field & Protective Cultivation***

Hydrogel is mixed with soil around the root zones of common trees and plants, providing water and nutrients stably over a period of time. While depending on young to fully grown trees, hydrogel amount differs from 20 – 100 gms mixed with the soil matrix per tree.

#### ***Terrace Farming, Home grown Gardens, and Vertical Farming***

Super Absorbent Polymer can be mixed with soil matrix, for indoor and open air pots, growers, window boxes, porches, terraces, hanging, greenhouses and city scene. Using SAP proves beneficial to water usage, time, labour and lessening drought and wilting risks which come into light if the plant is not watered for long, enabling a greener and bloomer period.

#### ***Arboriculture***

Arboriculture incorporates social techniques such as selection, planting, preparing, bug and pathogen control, pruning, molding, and evacuation. It is principally centered on individual woody plants and trees kept up for landscape and amenity purposes, ordinarily in gardens, parks, etc. under the

general umbrella of agriculture. Hydrogel reduces mortality rate due to transplantation shocks and enhances root development. A hole is dug about three times the volume of the root, at the plantation site, and 1 - 2 kgs of this product per m<sup>3</sup> of soil is mixed. The plant is placed the bottom of the hole and is evenly filled with the treated soil. The top surface is covered with 5 cm of untreated soil so as to prevent UV degradation of the product.

### ***Bare Root Dipping***

To prevent desiccation of the roots of seedlings during transplanting or transportation, 1 kg of potassium polyacrylate is mixed in 150 - 200 liters of water with/ without an additional fungicide/ bactericide, and allowing it to stand for 15 minutes. It finds a great use in flower preservation during transportation; fresher flowers which in turn increase their market value.

### ***Hydro Seeding***

Hydro seeding is a planting procedure that uses a mixture of seed and mulch, used as an erosion control system on construction destinations, and as another option to the conventional procedure of sowing dry seeds. Hydrogel mixed with cellulose mulch is used to stabilize newly graded soils, maintaining a minimum surface area, helping in sprouting of seedlings even in dry areas.

### ***Fertilizers***

Hydrogel polymers can be dry mixed into fertilizer preparations. It is able to absorb large volumes of liquids that can be released over a long time reducing leaching of essential soil nutrients. Therefore, this substrate was used for the slow release of fertilizers alone or in combination with a water-holding gel, dosed at 1 to 5kg by weight.

### ***Hydroponics/ Soil less media***

Hydroponics is a technique of growing plants using only mineral supplement solutions, in water, without soil. Plants might be grown with their roots in the mineral solution

only, or in a dormant/ inert medium. Hydrogel mixed with hydroponics media reduces the water stress.

### **1.2.3 Work principle of hydrogel**

Hydrogel polymer comes in crystal or powder, which jellifies upon contact with water or other liquids. These particles may be taken as mini water reservoirs in soil. It soaks up and stores water inside, with the capacity of absorbing nearly 400 – 600 times its own weight. Water from these reservoirs is released upon root demand through osmotic pressure difference. In arid areas, the use of hydrogel in sandy soils (macro porous medium) increases the water holding capacity, which significantly improves the quality of plants. Hydrogel Polymer can influence soil permeability, density, structure, texture, and evaporation rate of water through the soil. Hydrogel with excellent water absorption quality is an exceptionally helpful green and eco-friendly item for farming and agriculture fields. It can be used for farms and forestry services, sparing water for homestead and garden and enhancing trees' surviving ratio.

This aqua absorbent act as a reservoir of water and will only use the reserved resource at the time of need, allowing a better agricultural yield. Super Absorbent Polymers have a unique mechanism to absorb and retain water; discharging it only when the crop demands for it otherwise it does not lose out on the moisture level.

### **2.1 LITERATURE SURVEY**

The project is a basic try in the field of PLASTICULTURE where the plastic is being used in the field of agriculture to overcome the drawbacks. As in the introductory part, the mulching art is being incorporated with hydrogels for better water retention by crops.

Irrigation water is becoming scarce and the world is looking for water-efficient agriculture. Increasing food demand and declining water resources are challenges for food security (Kreye et al. 2009). With decreasing water availability,

rice production is needed to be switched towards water saving production systems. In the system of aerobic rice, especially adapted aerobic rice cultivars are grown under non-flooded or aerobic soils with supplementary irrigation (Bouman 2001, Bouman et al. 2005).

Hydrogel is a synthetic polymer, which is able to absorb and hold 80–180 times its volume of water for a long time (Wang and Gregg 1990). Hydrogel acts as a reservoir to store and release a steady stream of water and nutrients which plants need to grow. Plant roots are able to absorb water from the crystal bead of hydrogel. Several previous studies showed that these are very useful under limited water conditions to cope with plant water needs (Henderson and Hensley 1985, Ingram and Yeager 1987, Wang and Gregg 1990).

Johnson (1984) reported that addition of hydrogel at the rate of 2 g/kg improved the water holding capacity of sand from 171% to 402%. Application of hydrogel decreases the irrigation requirements of several crops by improving water holding capacity resulting in delay and onset of permanent wilting percentages under intense evaporation. An increase in water holding capacity due to hydrogel amendment significantly reduced the irrigation requirement of many plants (Taylor and Halfacre 1986).

Mulching reduces the deterioration of soil by way of preventing the runoff and soil loss, minimizes the weed infestation and checks the water evaporation. Thus, it facilitates for more retention of soil moisture and helps in control of temperature

fluctuations, improves physical, chemical and biological properties of soil, as it adds nutrients to the soil and ultimately enhances the growth and yield of crops. Further, reported that mulching boosts the yield by 50-60 per cent over no mulching under rain fed situations. (Patil shirish.S, 2013).

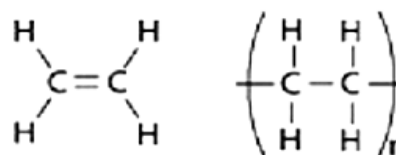
William (1993) reported that although a variety of vegetables can be grown successfully using plastic mulches. Muskmelons, honeydews, watermelons, squash, cucumbers, tomatoes, peppers, eggplant, okra, sweet corn, and cole crops have shown significant increases in earliness, total yield, and quality. Jaiswal et al. (1997) found that mulching of carrots was effective and root yield was increased by 34.6 % compared with no mulching. Islam et al. (2002) investigated the effect of planting time, mulching and irrigation on the growth and yield of cabbage cv. Atlas-70. Mulching and irrigation significantly affected the growth and yield of cabbage. The highest gross yield (71.85 kg/plot) was obtained from the black polyethylene mulch followed by water hyacinth mulch (65.99 kg/plot). Considering marketable yield, both black polyethylene mulch (103.01 t/ha) and water hyacinth mulch (90.99 t/ha) exerted statistically similar effects followed by irrigation at 15 days interval (85.85 t/ha), whereas non-mulching and non-irrigated plots (control) exhibited the lowest marketable yield (38.87 t/ha).

## EXPERIMENTAL WORK

### 3.1 Material Used

#### 3.1.1 Low Density Polyethylene

Low density polyethylene is a thermoplastic made from monomer ethylene. It is not reactive at room temperature, except by strong oxidising agents and solvents. It is flexible and tough and is either translucent or opaque. They are majorly semi crystalline. It contains the chemical components carbon and hydrogen.





Plastic film is a thin continuous polymeric material. These thin plastic membranes are used to separate areas or volumes, to hold items, to act as barrier or as printable surfaces. Plastic films are used in a wide variety of applications. These include: packaging, plastic bags, labels, building construction, landscaping, electrical fabrication, photographic film, film stokes for movies, video tape etc. On the other hand various colour mulches are produced based on their usage.

Generally the thickness of mulch does not majorly affect the efficiency of the mulch film. It's only the colour and additives that play a major role. But generally the thickness is in the range of 7 microns to 100 microns depending on the type of plant and its growth duration.

### Selection of mulch

The selection of mulches depends upon the ecological situations and primary and secondary aspects of mulching

Rainy season	Perforated mulch
Orchard and plantation	Thicker mulch
Soil solarisation	Thin transparent film
Weed control through solarisation	Transparent film
Weed control in cropped land	Black film
Sandy soil	Black film
Saline water use	Black film
Summer cropped land	White film
Insect repellent	Silver colour film
Early germination	Thinner film

**Table 1. Selection of mulch**

### 3.1.2 Carbon Black

Carbon black is an important and versatile ingredient for plastics compounders. It can contribute colour, opacity, electrical conductivity and protection from ultra-violet degradation. The choice of carbon black is dependent on the final product requirements. In this regard, particle size and structure (degree of permanent particle aggregation) are the two most important characteristics of a carbon black in determining its performance.

Carbon black is typically used in thermoplastics to impart at least one the properties below

- Colour
- UV Protection
- Conductivity

Since most thermoplastics are rigid at the end use temperature, the reinforcing effects of the carbon black have to be balanced with desired end use mechanical properties. In addition, the carbon black contaminants such as grit, ash and sulfur have a more crucial impact on the thermoplastic's mechanical properties and the processing of these materials. For plastics applications involving color and UV protection, carbon black is typically dispersed into a plastic master batch at a high dosage, 25 to 40% by weight.

The key performance criteria of carbon black products important to molded plastics applications are:

- Color Strength
- Blue Tone
- Effect on Master batch Viscosity
- UV Stability
- Dispersibility

### 3.1.3 HYDROGEL

The hydrogel was purchased from CHEMZEST Enterprises Pvt., Ltd. The hydrogel purchased was potassium based as it improves and stimulates the growth of the plant and also is non-toxic for the soil. The other type is sodium based but is said to damage the soil on continuous usage

and longer run. Thus the hydrogel purchased is “potassium polyacrylate”.

The hydrogel is directly available in the market is being used in the agricultural field. Thus this hydrogel was purchased to calculate its water absorbency. It was identified to absorb a minimum of 50% of its own weight and when left to dry it turned dark brown and shrank. Thus it does not damage the field when left to dry.

<b>Analysis</b>	<b>Unit</b>	<b>Specifica tion</b>
Appearance		White , granular
Particle size	Mm	Granular MS: 1.0- 2.0
Odor		Impercepti ble
Moisture	%	10 Max

Ph		7.0 – 8.0
Free Acrylamide content	Ppm	500 max
Bulk Density	g/cm <sup>3</sup>	0.80
Water Solubility		Insoluble
Toxicity In soil		None
Shelf life	Years	5
Degradability in Soil	Years	1-5
Absorbency	g/g	
Deionized Water		300-400
0.9% NaCl		100-150
Soil		150-200

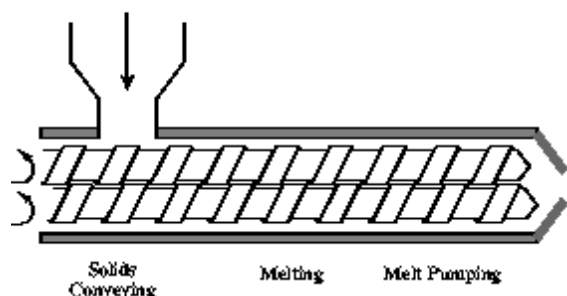
**Table 2 Specification of Hydrogel**

The hydrogel was heated upto 200°C in the hot air oven and then crushed to bring it to the powder form for easy miscibility. The crushed and powdered hydrogel was subjected to mesh analysis as to distinguish the sizes. The mesh numbers of mesh100, mesh200 and mesh350 was used to disintegrate the minimum of the size. The hydrogel was then stored in air tight packs for further preservation.

### **3.2 PROCESSING**

#### **3.2.1TWIN SCREW EXTRUSION**

Twin screw extrusion is used extensively for mixing, compounding, or reacting polymeric materials. The flexibility of twin screw extrusion equipment allows this operation to be designed specifically for the formulation being processed. For example, the two screws may be co-rotating or counter-rotating, intermeshing or non-intermeshing. In addition, the configurations of the screws themselves may be varied using forward conveying elements, reverse conveying elements, kneading blocks, and other designs in order to achieve particular mixing characteristics.



**Fig 2. Twin screw extrusion process**

The Low Density Polyethylene (LDPE) was blended with hydrogel using twin screw extrusion process. The temperature was maintained in a range of 110°C to 190°C and the composition of different components are as follows

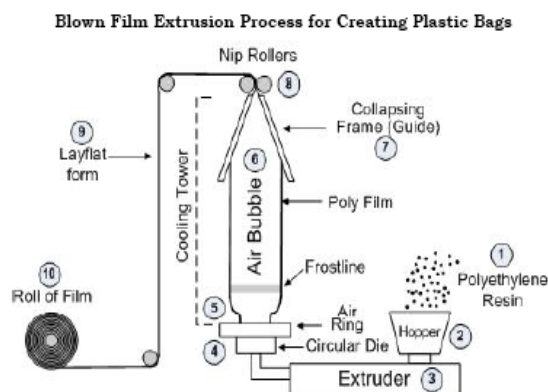
Components	Weight (gms)
LDPE Virgin granules	60
Potassium based hydrogel	10gms
Carbon black	gms
Compatibilizer	gms

**Table 3 Components used in extrusion process**

The extrudate was water cooled and then dried in hot air oven at 80°C for a period of half an hour. Thus, the collected extrudate was cut in small

sizes using a pelletizer. The pellets were then used in blown film extrusion to obtain a film.

### 3.2.2 BLOWN FILM EXTRUSION



**Fig 3 Blow film extrusion process**

The LDPE incorporated with hydrogel was air dried at 80°C for a period of 1 hour and then was extruded as film. The thickness of the film was maintained in the range of 1.5 – 2 cm. The temperature was maintained at 180°C. The obtained film was tested for color variation and presence of bubbles.

### 3.3 Testing Procedure

#### 3.3.1 Fourier Transform Infrared Spectroscopy (FTIR)

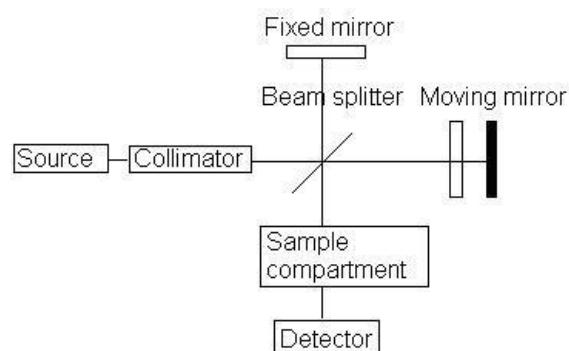
FTIR spectrometers (Fourier Transform Infrared Spectrometer) are widely used in organic synthesis, polymer science, petrochemical engineering, pharmaceutical industry and food analysis. In addition, since FTIR spectrometers can be hyphenated to chromatography, the mechanism of chemical reactions and the detection of unstable substances can be investigated with such instruments. The range of Infrared region is 12800 ~ 10 cm<sup>-1</sup> and can be divided into near-infrared region (12800 ~ 4000 cm<sup>-1</sup>), mid-infrared region (4000 ~ 200 cm<sup>-1</sup>) and far-infrared region (50 ~ 1000 cm<sup>-1</sup>). The discovery of infrared light can be dated back to the 19th century. Since then, scientists have established various ways to utilize infrared light. Infrared transform spectroscopy is the method

which scientists use to determine the structures of molecules with the molecules characteristic absorption of infrared radiation. Infrared spectrum is molecular vibrational spectrum. When exposed to infrared radiation, sample molecules selectively absorb radiation of specific wavelengths which causes the change of dipole moment of sample molecules. Consequently, the vibrational energy levels of sample molecules transfer from ground state to excited state. The frequency of the absorption peak is determined by the vibrational energy gap. The number of absorption peaks is related to the number of vibrational freedom of the molecule. The intensity of absorption peaks is related to the change of dipole moment and the possibility of the transition of energy levels. Therefore, by analyzing the infrared spectrum, one can readily obtain abundant structure information of a molecule. Most molecules are infrared active except for several homonuclear diatomic molecules such as O<sub>2</sub>, N<sub>2</sub> and Cl<sub>2</sub> due to the zero dipole change in the vibration and rotation of these molecules. What makes infrared absorption spectroscopy even more useful is the fact that it is capable to analyze all gas, liquid and solid samples. The common used region for infrared absorption spectroscopy is 4000 ~ 400 cm<sup>-1</sup> because the absorption radiation of most organic compounds and inorganic ions is within this region. FTIR spectrometers are the third generation infrared spectrometer. FTIR spectrometers have several prominent advantages: (1) The signal-to-noise ratio of spectrum is significantly higher than the previous generation infrared spectrometers. (2) The accuracy of wavenumber is high. The error is within the range of ± 0.01 cm<sup>-1</sup>. (3) The scan time of all frequencies is short (approximately 1 s). (4) The resolution is extremely high (0.1 ~ 0.005 cm<sup>-1</sup>). (5) The scan range is wide (1000 ~ 10 cm<sup>-1</sup>). (6) The interference from stray light is reduced. Due to these advantages, FTIR Spectrometers have replaced dispersive IR spectrometers.

## FTIR Spectrometers

### The Components of FTIR Spectrometers

A common FTIR spectrometer consists of a source, interferometer, sample compartment, detector, amplifier, A/D convertor, and a computer. The source generates radiation which passes the sample through the interferometer and reaches the detector. Then the signal is amplified and converted to digital signal by the amplifier and analog-to-digital converter, respectively. Eventually, the signal is transferred to a computer in which Fourier transform is carried out. **Figure 4** is a block diagram of an FTIR spectrometer.



**Figure 4. Block diagram of an FTIR spectrometer**

The major difference between an FTIR spectrometer and a dispersive IR spectrometer is the Michelson interferometer. Fourier transform infra-red spectroscopy (FTIR) is a technique which is used to obtain an infra-red spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. The goal of any absorption spectroscopy is to measure how well a sample absorbs light at each wavelength. The best method is by “dispersive spectroscopy” technique is to shine a

monochromatic light beam at a sample, how much of the light is absorbed and repeat for each different wavelength.

This FTIR was used to analyse the structural characteristics of the polyethylene film subjected to microbial action. The wave number ranges from  $400\text{ cm}^{-1}$  to  $4000\text{ cm}^{-1}$ . The films were analysed using NICOLET 6700 USA. A change in the structural characteristics was noticed that is discussed in results.

### 3.3.2 SCANNING ELECTRON MICROSCOPE (SEM)

#### Introduction:

The development of Scanning Electron Microscopy (SEM) in the early 1950's brought with it new areas of study in the medical and physical sciences because it allowed examination of great variety of specimens. As in any microscope, the main objective is magnification and focus for clarity. An optical microscope uses lenses to bend an electron beam, which is used to bend the light waves, and the lenses are adjusted for focus. In the SEM, electromagnets are used to bend an electron beam, which is used to produce the image on a screen. By using electromagnets an observer can have more control in how much magnification it obtains. The electron beam also provides greater clarity in the image produced. The SEM is designed for direct studying of the surfaces of solid objects. The SEM is a type of electron microscope capable of producing high-resolution images of sample surface. Due to the manner in which the image is created, SEM images have characteristic three-dimensional appearance and are useful for judging the surface structure of the sample.

SEMs are patterned after reflecting light microscopes and yields similar information:

**Topography:**The surface features of an object.

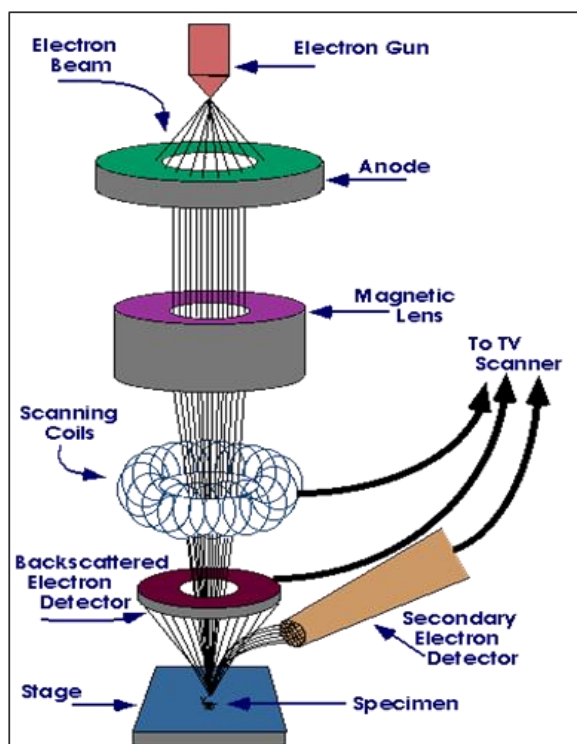
**Morphology:**The shape, size and arrangement of the particles making up the object that are lying on the surface of the sample.

**Crystallographic Information:**The arrangement of atoms in the specimen and their degree of order; only useful on single crystal particles >20 micrometers.

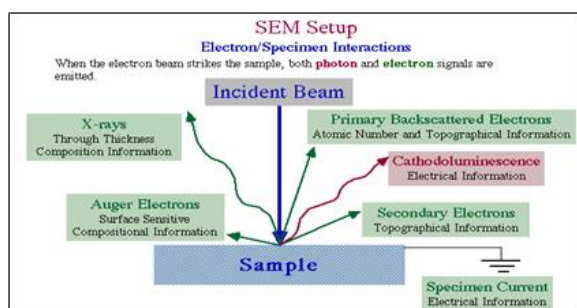
#### Working Principle:

The instrument can be simplified into three major sections: a) electron-optical 'column'; b) vacuum system and c) electronics and display system. A tungsten filament is heated to 2700 K, which produces electron gun, having stream of monochromatic electrons (energy ranging from a hundred eV to 50keV.) that are accelerated towards the anode disc. The stream is condensed by the first condenser lens to control the diameter and current of the beam. The beam is then constricted by the condenser aperture, eliminating some high-angle electrons. The second condenser lens forms the electrons into a thin, tight, coherent beam and usually controlled by the 'fine probe current knob'. A set of coils then scan or sweep the beam in a grid fashion dwelling on points for a period of time determined by the scan speed .

The final lens, the objective, focuses the scanning beam onto the part of the specimen desired. A cathode-ray display tube is scanned synchronously with the electron beam. The brightness of the display tube is modulated by the signal, which arises from the interaction of the beam with the surface element, which is probed. The strength of this signal is translated into image contrast. Secondary electrons, which the beam probe liberates from the specimen surface, are collected and used as the contrast signal. The yield of collected electrons depends on the nature of the specimen surface and on its inclination with respect to the probing beam. Consequently, one obtains pictures with a high perspective appearance.



**Fig 5 Schematic representation of SEM**



**Fig 6 SEM set-up electron specimen interaction**

Backscattered electrons produced by an incident electron colliding with an atom in the specimen, which is nearly normal to the incident's path. The incident electron is then scattered "backward" 180 degrees. The production of backscattered electrons varies directly with the specimen's atomic number or "specimen interaction volume". Specific interaction volume is the volume inside the specimen in which interactions occur while being struck with an electron beam. The differing production rates cause higher atomic number elements to appear

brighter than lower atomic number elements. This interaction is utilized to differentiate parts of the specimen that have different average atomic number. These elastically scattered electrons usually called 'backscattered electrons' are used for SEM imaging.

### Resolution of the SEM:

The spatial resolution of the SEM depends on the size of the electron spot, which in turn depends on both the wavelength of the electrons and the magnetic electro-optical system which produces the scanning beam. The resolution is also limited by the size of the interaction volume, or the extent to which the material interacts with the electron beam. The spot size and the interaction volume both might be large compared to the distances between atoms, so the resolution of the SEM is not high enough to image individual atoms, as is possible in the shorter wavelength (i.e. higher energy) transmission electron microscope (TEM). The SEM has compensating advantages, though, including the ability to image a comparatively large area of the specimen; the ability to image bulk materials (not just thin films or foils); and variety of analytical models are available for measuring the composition and nature of the specimen. Depending on the instrument, the resolution can fall somewhere between less than 1 nm and 20 nm. Resolution is 0.4nm at 30Kv and 1.6nm at 1Kv. In general, SEM images are easier to interpret than TEM images.

### Test Procedure:

In the present investigation the morphology of the nanocomposites was observed using SEM-JSM-6390 (JEOL Ltd, Japan) with 15 kV accelerating voltage at 5mm resolution. Specimens are platinum coated at low temperature. Ultra-thin specimens of 100 $\mu$ m thickness were cut from fractured surface of tensile specimen using Reichert ultra-cut microtome. The specimens were collected on a

trough filled with water and placed on a 200 – mesh grids.

Scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. SEM was used to analyse visually the changes on the surface of the polyethylene film subjected to microbial action. Polyethylene being non-conducting in nature was subjected to gold sputtering before subjecting it to the action of electron beams. The magnification in the range of 1000X to 4000X was analysed. Significant changes were observed on the surface of the polyethylene film which proved microbial degradation.

### 3.3.3 WATER ABSORPTION TECHNIQUE

The test was carried out using the procedure ASTM D570. Moisture absorption (also known as water absorption) is the capacity of a material to absorb moisture from its environment. Plastics absorb water to a limited degree. The degree of moisture absorption depends on the type of plastic and the ambient conditions such as temperature, humidity and contact time. Not only can dimensions change due to moisture absorption, but also material properties, such as mechanical strength, electrical conductivity and the dielectric loss factor, can be also affected.

The moisture absorption leads to changes in dimensions of finished products, a reduction in strength and also changes in electrical insulating characteristics. Various polymeric materials are susceptible to water absorption during its life exposure. This may cause dimensional instability with property degradation and ultimately lead to failure. This test was designed to evaluate materials by exposing specimens to water for different time & temperature profiles. Testing is conducted on specimens that are submersed in water and a before & after weight change is documented. Depending on the application of the product, certain levels of moisture are accepted.

Certain materials require small amounts of moisture for ultimate field performance. However, it is generally accepted that the lower the number, the better, especially during molding or extrusion. Most polymers are dried @ 500°C in an Air Circulated Oven for 24 hours and re-weighed to the nearest 0.001 gms. High Engineered grades (filled or hi-temp.) may require higher drying profiles, but the time is generally the same.

Polyamides (nylons) generally show higher water absorption than other engineering plastics. The only polymer with zero water absorption is PTFE. Plastics with very low water absorption are PEEK, PPS, PPSU, PVDF, PET, PPE, PP and PE. Furthermore, low water absorption is exhibited by POM, PA12, PC and ABS.

The result shall be either increase or decrease in weight depending on the composition and types of ingredients being used. The results are calculated as follows

$$\text{Increase in weight \%} = \frac{\text{Wet weight} - \text{Conditioned weight}}{\text{Conditioned weight}} \times 100$$

In case of soluble substances being present the weight decreases and the result is calculated using the formula,

$$\text{Soluble matter, lost \%} = \frac{\text{Conditioned weight} - \text{Reconditioned weight}}{\text{Conditioned weight}} \times 100$$

### 3.3.5 DENSITY



**Fig 9 METTLER TOLEDO weighing balance**

Density is the mass per unit volume of a material. Specific gravity is a measure of the ratio of mass of a given volume of material at 23°C to the same volume of deionized water. Specific gravity and density are especially relevant because plastic is sold on a cost per pound basis and a lower density or specific gravity means more material per pound or varied part weight. There are two basic test procedures- Method A and Method B. The more common being Method A, can be used with sheet, rod, tube and molded articles. For Method A, the specimen is weighed in air then weighed when immersed in distilled water at 23°C using a sinker and wire to hold the specimen completely submerged as required. Density and Specific Gravity are calculated. The density measurement was done following the ASTM D792 method. The METTLER TOLEDO analytical and precision balance was used to perform the density measurement. LDPE having density lesser than that of water, benzene was used as the liquid medium. The density was calculated using the formula

$$\text{Density(g/cc)} = \frac{\text{Weight in air} - \text{Weight in liquid}}{\text{Weight in liquid}} \times \text{density of liquid}$$

The major advantages of the machine are

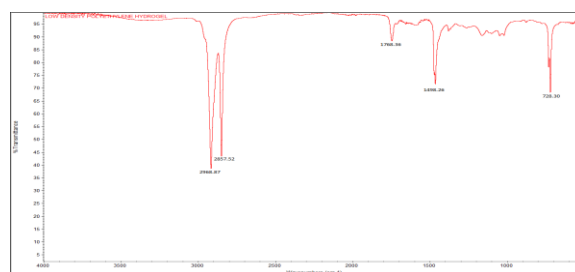
- High flexibility to accommodate individual process needs
- Automatic density calculations for solids and liquids including temperature adjustment of the reference liquid
- Statistic evaluation of multiple samples
- All results, including User, Sample ID, Lot Number, Time and Date can either be printed or saved on a USB stick.

Thus, using density test method, the variations in density can be observed and further improvisation required can be altered in materials as such.

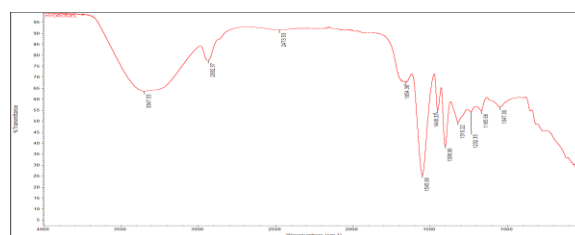
## RESULTS AND DISCUSSIONS

### 4.1 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a technique used to obtain an infra-red spectrum of absorption. This FTIR was used to analyse the structural characteristic of the films subjected to microbial action. The FTIR was taken for both low density polyethylene (LDPE) and hydrogel. The results obtained are as follows



**Fig 4.1 The FTIR spectrum of LDPE**

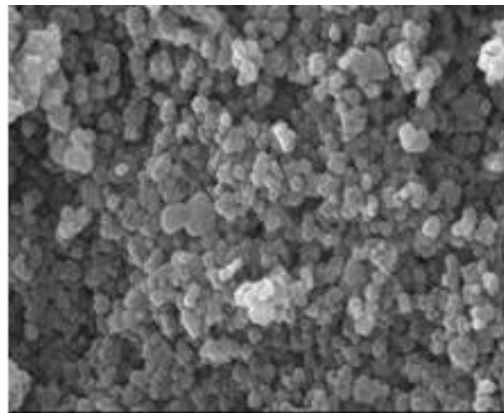
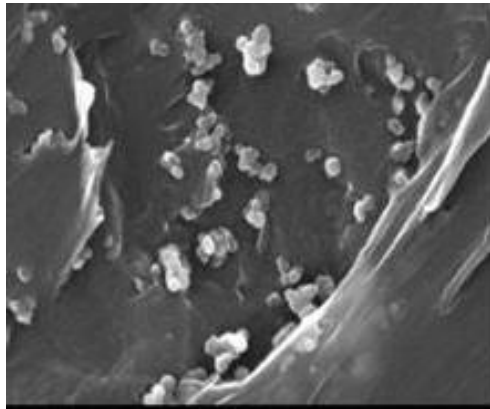




**Fig 4.2 The FTIR spectrum of hydrogel**

#### **4.2 SCANNING ELECTRON MICROSCOPE (SEM)**

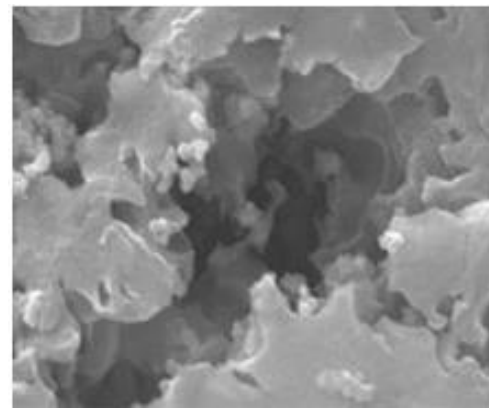
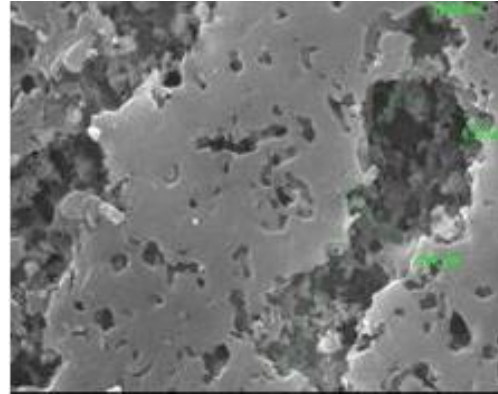
The SEM was performed to analyse the structural changes on the surface of the film. The test was performed based on before and after usage in soil. The results are observed as follows



**Fig 12 The SEM micrograph of LDPE with hydrogel after blending process (a) 15000X (b) 20000X**

The film was then subjected to continuous alternate sunlight and water as in case of agriculture. The film was then subjected to SEM analysis to observe the changes in the surface. It was clearly seen the hydrogel particles been

eroded and the film was cracked open in several areas indicating the hydrogels being dissolved into the soil surface. The obtained results are as follows



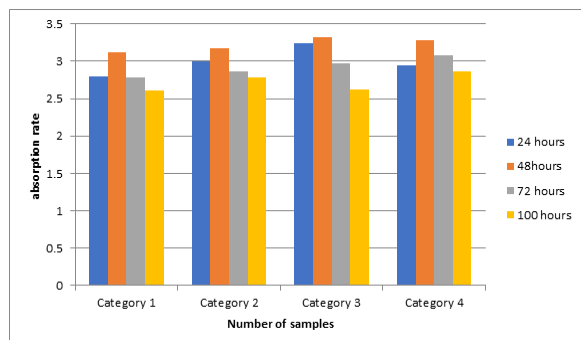
**The SEM micrograph of LDPE with hydrogel after usage as mulch film (a) 15000X (b) 20000X**

This supports the fact that weight loss being observed in the water absorption analysis. Thus, the hydrogel particles have got lossen away from the LDPE film surface and thus leaving holes and cracks on them. This clearly indicates that hydrogel being mixed with the soil and acts as water storage units.

#### **4.3 WATER ABSORPTION ANALYSIS**

For this technique the film was cut into small pieces of 5X5 cm and weighed before being

suspended in water. The test was carried out in four different ceramic bowls and weight analysis was carried out in time interval of 24 hours for seven days. The results are as follows



The results observed was that initially there was an increase in weight, but after 48 hours the weight dropped down on daily basis and at the end of 100 hours a constant weight was observed. This indicates that initially, the water was being absorbed by the hydrogels been present in the film, which caused an increase in weight. Later, with the proof of SEM analysis, the hydrogel particles were seen to drift away from the film and been mixed with water.



**Fig 14 Weight analysis of four different samples**

The same analysis was done using soil instead of water, as in case of being used in agriculture. The film was cut to a size of 10X5 cm and a plastic bowl was taken. Two such bowls were filled with

soil and the soil was covered with mulch film. On a daily basis, the soil was watered every day and weight analysis was done to the film. The same was observed where the weight of the film increased on 24 hours basis and later decreased gradually indicating loss of hydrogel particles.



**Fig 15 The mulch film with soil for weight analysis**

The changes in weight was observed as following

samples	4 hours	8 hours	2 hours	00 hours
sample 1 (10 gms)	1.28	0.54	0.86	0.2
sample 2 (10 gms)	1.62	1.06	0.84	0.68

**Table 4 Changes in weight on water absorption**

Thus, the samples weight decreased and was seen to be constant after a span of 100 hours. The both samples were subjected to SEM analysis to observe the morphological changes.

#### 4.4 DENSITY

As LDPE, having a density lesser than water, benzene was used as medium having a density of 0.875 g/cc. The test was carried out using

METLER TOLEDO analytical balance. The results obtained are as follows

Samples	Density (g/cc)
Sample 1	0.948
Sample 2	0.942
Average	0.945

**Table 5 Density variation**

Thus, the density has slightly increased when compared to LDPE virgin film being only 0.938 g/cc. Thus, the hydrogel have contributed to an instant increase in weight.

## CHAPTER V

### CONCLUSION

The LDPE was blended with hydrogel using twin screw extruder at around 180°C and then film was extruded using blow film extrusion at a temperature of about 200°C. The film was then subjected to water absorption using four different samples and an average rate of absorption was calculated. Then the film was subjected to coverage with soil as in case of being used in agriculture.

The hydrogels were seen to dissolve into the soil, as a result the water absorption was increased. The results were proven using changes observed in morphological unit using Scanning Electron Microscope (SEM), Water absorption technique and density. The FTIR result was taken for both hydrogel and Low density Polyethylene to identify the miscibility possibilities. Also, by capillary method, the melting point for hydrogel was calculated. It did not melt or degrade till a temperature range of 340°C. Only change is the colour turning from white to light brownish shade.

The scanning electron microscope (SEM) proved the miscibility of Low

density polyethylene with that of potassium based hydrogel. The hydrogel being in nano particle size, they were blended with LDPE easily and thus, this has been proven by the magnification. On usage with soil, the film was washed and analysed further, the disappearance of hydrogels were noticed and further the cracks have been developed on the surface of the film. Thus, this proves that hydrogels has been leached out on to the soil.

The water absorption technique proves the rate of water absorption. The average percentage of absorption is around 26% and drip irrigation method has been used. But the fluctuations in absorption has also been observed. This technique further analyses that using different types of irrigation the absorption rate varies.

The density test was made to analyse the changes in density of the mulch film. The film showed an increase in density as compared to that of virgin Low density polyethylene. Also, morphological changes were noticed. The film was easily ruptured, hence mechanical analyses were difficult to be made. Also, the film has to be degraded after a period of 6 months, hence analysis were made by blending mulch film with that of poly lactic acid to make it degradable easily.

Thus, when being blended the hydrogels shows a higher rate of disposal into the soil when compared to that of being dispersed directly into the soil. Also by this method, the consumption of hydrogels can be effectively increased. Thus, this helps in saving of water consumption and that the calculated amount of water absorption takes place.

## CHAPTER VI

### FUTURE WORK

Though the potassium based hydrogel incorporated mulch film proves a boon on water storing capacity, the major concern relies on the disposal after usage. Once, the harvesting period is over, the mulch film has to be removed and replaced with a new one. The problem arises with that of degradation. So, the future work has to be concentrated on making it more of bio-degradable after serving its purpose.

Also, on degradation, the film should either provide nutrients to the soil or on other hand should not cause soil pollution. Thus, the following has to be improvised on the next stage

- The mulch film on disposal should not create soil pollution.
- The film should be made bio-degradable.
- On being bio-degradable, the degradation should start only after harvest period and the ingredients should not affect the plant growth.

Thus, the further improvisation shall lead to more of commercialization with further improved properties.

## CHAPTER VII

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