Crop Response to Water and Fertilizers used in Soil modified with Hydrogels

By

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Dedication

This thesis is dedicated to my lovely parents Mrs. Ruchi and Mr. Hitesh, my dear siblings Saksham and Aayushi.

Abstract

Increasing water and nitrogen use efficiency is essential to increase crop production and to reduce environmental degradation. Cellulosic hydrogels derived from paper waste, have the ability to retain and gradually release water and nitrogen for optimum plant growth. This study assessed crop response to water and $NO_3^- - N$ with hydrogels amended in the soil. Tomato was used as the crop for this study as it is the most widely cultivated vegetable crop with a sensitive response to water and nitrogen. An experiment was conducted during 2020-2021 in the research greenhouse at Macdonald Campus of McGill University, comprising the following treatments: freeze-dried hydrogels (FDH), oven-dried hydrogels (ODH), control (without hydrogels) as well as two irrigation treatments (95% and 75% available water content (AWC)). Equivalent beads (32.10 of FDH and 35.96 g of ODH) corresponding to 4.6 g of 20-20-20 N-P-K fertilizer were applied before transplanting, at a depth of 0.15m from the soil surface. The treatments were replicated three times using a factorial design. The results indicated that FDH- 95% AWC treatment produced the highest average crop yield of 0.88 kg plant^{-1}, compared to the ODH (0.32 kg plant^{-1}) and control treatments (0.40 kg plant^{-1}). The hydrogel and AWC combinations did not significantly ($p > 0.05$) impact plant height and stem diameter, while these treatment combinations enhanced and significantly affected crop yield, leaf area index and plant biomass ($p < 0.05$). FDH and ODH produced a substantially higher yield and saved 15 % and 20% of irrigation water (225mm) as compared to the control treatment. Furthermore, there was a noticeably higher water use efficiency in the FDH-95 (3.911 kg m^{-1} plant^{-1}) treatment as compared to the ODH-95 (1.467 kg m^{-1} plant^{-1}) and control (1.509 kg m^{-1} plan^{-1}) treatments. With soil only, and no crop, FDH was most effective in releasing fertilizer to the plants. The FDH gradually increased $NO_3^- - N$ concentration from 20 to 65 mg kg^{-1} over a month. The results indicate that under FDH and ODH treatments, excess nitrate was stored in the soil vacuoles, and was remobilized for uptake by the plant roots. The overall performance of both hydrogels was comparatively better than the control, with the FDH-95% AWC giving the highest marketable yield, and best water and nitrogen saving potential. This study showed that cellulose -paper-based hydrogels, which is a waste product from the pulp and paper industry, can be used to improve crop production.

Résumé

L'augmentation de l'efficacité de l'utilisation de l'eau et de l'azote est essentielle pour augmenter la production agricole et réduire la dégradation de l'environnement. Les hydrogels cellulosiques dérivés de déchets de papier, ont la capacité de retenir et de libérer progressivement l'eau et l'azote pour une croissance optimale des plantes. Cette étude a évalué la réponse des cultures à l'eau et au NO3−−N avec des hydrogels amendés dans le sol. La tomate a été utilisée comme culture pour cette étude car c'est la culture légumière la plus largement cultivée avec une réponse sensible à l'eau et à l'azote. Une expérience a été menée en 2020-2021 dans la serre de recherche du campus Macdonald de l'Université McGill, comprenant les traitements suivants : hydrogels lyophilisés (FDH), hydrogels séchés au four (ODH), contrôle (sans hydrogels) ainsi que deux irrigations traitements (95% et 75% de teneur en eau disponible (AWC)). Des billes équivalentes (32,10 de FDH et 35,96 g d'ODH) correspondant à 4,6 g d'engrais 20-20-20 N-P-K ont été appliquées avant le repiquage, à une profondeur de 0,15 m de la surface du sol. Les traitements ont été répliqués trois fois à l'aide d'un plan factoriel. Les résultats ont indiqué que le traitement FDH-95% AWC produisait le rendement moyen le plus élevé de 0,88 kg plante-1, par rapport à l'ODH (0,32 kg plante-1) et aux traitements témoins (0,40 kg plante-1). Les combinaisons hydrogel et AWC n'ont pas eu d'impact significatif ($p > 0,05$) sur la hauteur des plantes et le diamètre de la tige, tandis que ces combinaisons de traitement ont amélioré et affecté de manière significative le rendement des cultures, l'indice de surface foliaire et la biomasse végétale ($p < 0,05$). FDH et ODH ont produit un rendement sensiblement plus élevé et économisé 15 % et 20 % d'eau d'irrigation (225 mm) par rapport au traitement témoin. En outre, il y avait une efficacité d'utilisation de l'eau sensiblement plus élevée dans le traitement FDH-95 (3,911 kg m-1 usine-1) par rapport à l'ODH-95 (1,467 kg m-1 usine-1) et au contrôle (1,509 kg m- 1 plan-1) traitements. Avec du sol uniquement et sans culture, le FDH a été le plus efficace pour libérer les engrais sur les plantes. La FDH a progressivement augmenté la concentration de NO3−−N de 20 à 65 mg kg-1 sur un mois. Les résultats indiquent que sous les traitements FDH et ODH, l'excès de nitrate a été stocké dans les vacuoles du sol et a été remobilisé pour être absorbé par les racines des plantes. La performance globale des deux hydrogels était comparativement meilleure que celle du contrôle, le FDH-95 % AWC donnant le rendement commercialisable le plus élevé et le meilleur potentiel d'économie d'eau et d'azote. Cette étude a montré que les hydrogels à base de cellulose et de papier, qui est un déchet de l'industrie des pâtes et papiers, peuvent être utilisés pour améliorer la production agricole.

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List of Abbreviations and Acronyms

%	percent	NC	Nitrate content
2M KCl	A solution of KCl bearing 2 moles KCl L^{-1}	NUE	Nitrogen use efficiency
AAS	Atomic absorption spectrophotometer	NH_4^+	Ammonium ions
AWC	Available water content ($\psi_{fc} - \psi_{pwp}$)	NO_3^-	Nitrate ions
DV	Daily value	OD	Oven-dried
ECH	epichlorohydrin	ODH	Oven-dried hydrogels
FAO	United Nations Food and Agriculture Organization	OP	Office paper
FC (θ_{fc})	Soil field capacity	P	Phosphorous
FD	Freeze-dried	PWP (θ_{pwp})_	Soil permanent wilting point
FDH	Freeze-dried hydrogels	RD	Rooting depth
HP	Hydrolyzed paper	RETC	Retention curve
ISE	Ion-selective electrode	SAP	Superabsorbent polymers
ISFET	Ion-selective field-effect transistor	SP	Shredded paper
IWUE	Irrigation water use efficiency	SWC (θ)	Soil water content
K	potassium	TAW	Total available water ($\psi_{fc} - \psi_{pwp}$)
LAI	Leaf area index	TDR	Time domain reflectometry
m.c.	Moisture content	WUE	Water use efficiency
MAD	Manageable allowable depletion		
N	Nitrogen		

Chapter 1: General Introduction

1.1 Background

Over the last few decades, global population growth has led to increased food demands. This increase in food production requires more efficient use of water and crop nutrients. Water scarcity is exacerbated by droughts, arising from climate change, and is expected to worsen in the future (Jovanovic et al., 2020; Pachauri et al., 2014; Spinoni et al., 2020). Sustainable management practices are required to make more equitable use of limited natural resources such as water and nutrients.

Globally, the use of inorganic nitrogen fertilizers for various forms of crop production has been steadily increasing since the onset of the Green Revolution. Although the application of nitrogen fertilizers contributes to crop yield improvement, their over-utilization can cause severe threats to water quality (Ahmed et al., 2017). Therefore, there is a need to develop and assess more innovative and sustainable methods of utilizing agricultural inputs (Boretti and Rosa, 2019).

One of the soil amendments that can be used to maximize soil water availability for plants is the use of hydrogels. They broadly enhance soil water holding capacity, hence increasing water and nutrient use efficiencies. Hydrogels are a water-swollen cross-linked polymer, possessing a water-absorbing capacity of 10 to 100-fold greater than their dry mass, distributed in their three-dimensional networks (Ahmed, 2015; Li and Chen, 2019; Nascimento et al., 2018).

The use of hydrogel amendments in crop production can help in curtailing water loss during irrigation. Hydrogels can also increase crop production by timely releasing nutrients directly into the root zone during the growing season. While applicable to all regions, they are most effective in arid and semi-arid regions. Hydrogels can minimize irrigation frequency, provide better oxygenation to the plant roots, delay the dissolution of fertilizers, and increase plant growth (Guilherme et al., 2015).

A wide range of techniques can be employed to manufacture hydrogels. These include single-step procedures such as polymerization and parallel cross-linking of multifunctional monomers, and multi-step procedures such as polymer synthesis and successive cross-linking, by reacting polymers with suitable cross-linking agents. Hydrogels can be natural or synthetic depending on the materials used for production (Ahmed, 2015).

Most synthetic hydrogels are non-biodegradable, as they are manufactured using acrylate polymers, which disintegrate very slowly, leading to environmental pollution (Abobatta, 2018). Therefore, in the present study, we used natural-based cellulose hydrogels, originating from cellulose waste, which is an underutilized source that can offer environmental as well as economic benefits (Durpekova et al., 2020; Mali et al., 2018; García et al., 2020).

1.2 Overall Objectives

The overall objective of the research was to assess crop response to water and fertilizers applied to soils amended with hydrogels.

1.2.1 Specific Objectives

The overall objective is achieved through the following specific objectives:

i. To estimate fertilizer decay pattern in the presence and absence of a crop grown under different hydrogel and water treatment levels.
ii. To assess crop response to two types of hydrogels (freeze-dried and oven-dried).
iii. To determine irrigation water-use efficiency of the hydrogels, under a tomato crop.

1.3 Scope

The application of cellulose hydrogels on mineral soil will benefit crop growers by reducing water losses during irrigation and improving nutrient uptake efficiency. The findings obtained in this study apply to tomatoes grown in a controlled environment in the greenhouse. Extrapolating the findings to other crops and growing conditions should be done with caution and professional discretion.

Chapter 2 : Literature review

2.1 Water scarcity

Freshwater makes up around 0.01 % of the total amount of water on the planet. Because of the population explosion and industrial growth in the nineteenth and twentieth centuries, freshwater consumption increased dramatically. Up to two-thirds of the world's population is expected to live in water-stressed countries by 2025, with water availability of less than 1000 m^3 per capita (Dimkpa et al., 2017) and the situation is likely to worsen by 2050.

Agriculture is the world's largest consumer of water (FAO, 2015), accounting for 65 % to 75 % of all freshwater use (Ali et al., 2020). Pressures on agricultural production are increasing as the world's population grows (Chaudhary and Srivastava, 2021). Furthermore, water consumption is projected to be 55% higher by 2030 compared to the consumption in 2005, and agricultural water withdrawals may increase by 66% compared to the year 2000 (Suresh et al., 2018). Excess nutrients in drainage from agricultural areas, particularly Nitrogen (N) and Phosphorus (P) contribute to eutrophication and cyanobacterial blooms in surrounding water bodies, resulting in loss of biodiversity, negative socioeconomic effects, and environmental concerns (Madramootoo et al., 2021).

2.2 Plant response to water stress

In most herbaceous plants, water accounts for 80-90 % of the total weight. Therefore, adequate soil available water content is vital for optimum plant growth (Kramer and Boyer, 1995). Inadequate water availability affects around one-third of the world's arable land, resulting in lower yields owing to drought during the agricultural season (Singh et al., 2016). Water scarcity has a significant influence on the root-shoot ratio at the plant level and increased mechanical impedance which reduces plant growth rates dramatically (Zhang et al., 2021). Furthermore, reduced permeability caused by root suberization, and the loss of fine roots might impair the balance between water extraction capability and transpiring leaf area as the soil dries (Akıncı and Lösel, 2012).

Water stress and irrigation scheduling threshold levels can be determined using a variety of methods. The methods range from estimating a classical water balance to soil moisture-based methods, reflecting plant responses to water stress (Niinemets, 2010). Soil moisture status, plant–root interface water uptake capacity, internal hydraulic conductivity, and evaporative demand all have a cumulative effect on plant growth parameters that can be measured by plant-based techniques (Parkash and Singh, 2020).

Leaf wilting reduced leaf area and stem diameter growth, as well as changes in biophysical processes like photosynthesis, stomatal conductance, leaf water status, and osmotic adjustments are all caused by reduced evapotranspiration. Two of the most important plant-based water-stress indicators are the potential and the relative water content of the leaf (Aknci and Lösel, 2012; Osakabe et al., 2014).

Stomata has a crucial function in the soil–plant–atmosphere continuum to regulate water flow. For different soil moisture and climatic conditions, stomatal changes aids in controlling plant water status. Greater stomatal conductance, the physical impedance to gas flow between the leaf interior and the atmosphere through the stomatal aperture, aids CO_2 absorption and water losses (transpiration) (Damour et al., 2010). Both processes of transpiration and gas exchange are essential for the plant's normal operation.

Transpiration aids in the regulation of leaf temperature, which is essential for the plant's metabolic activities. Stomatal conductance decreases in water-stressed conditions as stomata close to maintaining leaf water status (Chaves et al., 2003). Chemical signals are thought to be responsible for stomatal closure in some studies, while hydraulic signals are thought to be responsible in others. Furthermore, water deficiencies can arise not only in arid and semi-arid but is also prevalent in tropical rainforests (Akıncı and Lösel, 2012).

2.3 Use of fertilizer in agriculture

Fertilizers enhance crop yield by providing a specific blend of water-soluble plant nutrients. Blended inorganic fertilizers play a crucial role in the world's food security as they support the production and optimization of all cropping systems (Stewart and Roberts, 2012).

The key advantage of inorganic fertilizers over organic fertilizers is that these rich nutrient salts dissolve rapidly and are readily available to the plants. The 4Rs (right source, right rate, right time, and right place) are the underlying principles of fertilizer management (Johnston and Bruulsema, 2014). Therefore, appropriate management techniques should be implemented for the effective application of fertilizers for crop production.

The composition of N-P-K fertilizer often consists of ammonia, ammonium phosphate, superphosphates, Urea, Muriate of potash, and other microelements. A balanced proportion (1:1:1) of N-P-K makes it a versatile fertilizer and aids in fertilizing plants at all stages of growth. These NPK fertilizers act as a catalyst for crop growth to their full potential and provide resistance against diseases and various pests (Bergman, 1989).

2.4 Crop response to nutrients

Nutrient deficiencies have a significant impact on agriculture, resulting in decreased crop yield or plant quality (Morgan et al., 2013). Plant growth and development are heavily influenced by the concentration of mineral nutrients in the soil. Plants require 14 nutrients in adequate quantities to meet the demands of basic cellular functions and provide optimum yield (Havlin, 2020). Nutrient deficiencies can lead to reduced production of chlorophyll, a pigment required for photosynthesis, can cause stunted growth and plant tissue death (McCauley et al., 2009).

Nutrient management is essential for maintaining sustainable crop yields. Each nutrient plays a vital role in plant growth and development. Elements needed in larger amounts by the plant are referred to as macronutrients while the elements needed in lower quantities and whose deficiencies are equally damaging to plant growth in terms of yield and profit are termed micronutrients. Nitrogen (N), Phosphorous (P), and Potassium (K), Calcium (Ca), Sulfur (S), and Magnesium (Mg) fall under the category of macronutrients while Iron (Fe), Manganese (Lim et al.), Boron (B), Zinc (Zn), Copper (Cu), and Molybdenum (Mo) (n.d.) are classified as micronutrients (Jat et al., 2015).

Table 2.1: Essential plant nutrients with the absorptive chemical forms, roles, and deficiency causes

Element	Chemical symbol	Form of absorption	Role	Deficiency symptoms
Nitrogen	N	Nitrate (NO_3^-), Ammonium (NH_4^+)	Builds protein and leaf growth; found abundantly in all plant cells, easily leaches from the soil.	General discoloration and yellowing of leaves, leading to stunted plant growth.
Phosphorous	P	Dihydrogen phosphate ($H_2PO_4^-$, HPO_4^{2-}), Phosphate (PO_4^{3-})	Important for root development builds membranes and allows the plant to transfer energy from the sun, stays in soil longer.	Older leaves turn dark green or reddish-purple, leading to dwarfed or stunted plants.
Potassium	K	Potassium ion (K^+), Muriate of potash.	Maintains water and salt balance, helps plants tolerate the drought, heat and be disease resistant, but can be easily leached from the soil.	Wilted leaves, yellowing between veins, dead spots on older leaves.

Source: (Solution Center for Nutrient Management, 2021)

2.5 General introduction on hydrogels

Innovation in the polymer industry, known as hydrogels, provides high water and mineral retention capacity (Singh et al., 2021). Hydrogels, commonly defined as a network of hydrophilic polymers, that can swell and hold a large amount of water, were first reported by Wichterle and Lim in the 1960s (Ahmed, 2015). There must be a water content of at least 10% of the total weight of the polymer material, for it to be considered as a hydrogel. Various hydrophilic groups are attached to the polymer network, primarily, $-NH_2$, -COOH, -OH, $-CONH_2$, -CONH, $-SO_3H$ conferring the relative hydrophilicity of the network. In our daily lives, sponges and paper towels are often used to absorb water, yet, due to their low water retention qualities, these materials absorb only small amounts of water (Liu and Rempel, 1997). In contrast, superabsorbent polymers (SAP), may hold a large amount of liquid relative to their mass (Suresh, 2015). Hence, SAP's are the best-suited materials to be used in dryland agriculture, which can increase water and mineral retention capacities.

One of the most promising options in super absorbent polymers is the injection of fertilizers into the hydrogel beads. It can be used to apply mineral fertilizers to crops along with irrigation water. It is an efficient way to satisfy plant demand for nutrients from the control volume by synchronizing the supply of water and nutrients during the growth stages of a specific crop (Kafkafi and Kant, 2005).

The regulated release of nutrients from superabsorbent hydrogels based on polysaccharides, (i.e., chitosan, pectin, carboxymethyl cellulose) allows them to act as carriers to fertilize the soil. Their use will further minimize the nutrient leaching losses and will decrease the environmental footprint to anthropogenic agricultural practices (Elbarbary and Ghobashy, 2017). Various properties of conventional and hybrid hydrogels are mentioned in Table 2.2.

Table 2.2: Properties of conventional and hybrid hydrogels (Simoni et al., 2017).

Physical properties	Conventional		Hybrid
Source	Natural	Synthetic	Natural combined with synthetic
Cross-linking	Physically linked		Chemically linked
Degradability	Biodegradable		Non-biodegradable
Preparation	Copolymeric	Homo-polymeric	Interpenetrating network
Ionic charge	Cationic	Anionic	Non-ionic
Response	Physical agents: temperature, pressure, light, electric field, magnetic field.		Chemically responsive: biochemical agents, antigens, enzymes, ligands

2.5.1 Types and preparation of hydrogels

Hydrogels take water in through a diffusion-driven mechanism that depends on a gradient, *i.e.*, the presence of a moisture gradient between inside the hydrogel and water outside the hydrogel (Mohamadnia et al., 2008). When the hydrogel polymers are placed in water, water diffuses into the polymeric hydrogel via the process of osmosis. During this process, the H^+ atoms come out and negative ions are left behind along the polymeric chain. Several negative charges, due to the same ion strength, repel each other. Due to this resistive force, the polymer chain is forced to unwind and hence, attracts the H_2O molecules, primarily by hydrogen bonding. The three integral components of the hydrogel preparation are monomer, initiator, and crosslinker (Štular et al., 2017). The purpose of adding crosslinker material is to interconnect molecules, improve the properties of hydrogels, to build their 3D structure thereby enhancing their molecular weight, which improves its mechanical capabilities, and affects physical parameters including polymer elasticity, viscosity, and insolubility (Reddy et al., 2015; Sirajuddin et al., 2014).

Hydrogels are usually prepared from polar monomers. Based on their configuration which arises from their physical structure and method of preparation, hydrogels can be classified as amorphous, crystalline, or semi-crystalline (Karunarathna et al., 2019; Miyata et al., 2002). The four groups involved in the formation of bonds include non-ionic hydrogels, ionic hydrogels, amphoteric electrolytes, and zwitterionic hydrogels (Behera and Mahanwar, 2020). According to their origin, they can be divided into natural and synthetic polymer hydrogels (Chang et al., 2010).

2.5.1.(a) Natural hydrogels

Natural hydrogels are often based on polysaccharides or protein chains. Polysaccharides have an excellent ability to form hydrogels due to their hydrophilic structure. Some polysaccharides, such as starch, cellulose, sodium alginate, chitosan, guar gum, carrageenan, and others, are often used as natural hydrogels in the preparation of environmentally friendly hydrogels. Due to several advantages offered by cellulose and its derivatives, it is regarded as a hydrological standout (Simoni et al., 2017).

Cellulose-based hydrogels can be manufactured in two ways: 1) chemical crosslinking, where cellulose or cellulose derivatives are crosslinked via a di-functional molecule (i.e., epichlorohydrin (ECH) (Zhou et al., 2007); and 2) physical crosslinking, where non-derived cellulose can be crosslinked through rearrangement of their intra- and intermolecular hydrogen bonds (Wang and Chen, 2011). Due to the various hydrophilic hydroxyl groups attached to the cellulose molecular chains forming a permeable structure, cellulose hydrogels possess the potential to absorb and store a substantial volume of water in their three-dimensional matrix (Li and Chen, 2019; Nascimento et al., 2018). Peng et al. (2012, 2013) evaluated various drying processes for obtaining cellulosic hydrogel structures: a) Freeze Drying (Lyophilization) b) Oven Drying (Solvent Evaporation) c) Spray Drying d) Supercritical Drying (Zimmermann et al., 2016).

The key techniques of water removal are room temperature/air drying and oven drying (OD).

However, there is a chance of losing the original porous microstructure from the moist materials during these processes (Pa’e et al., 2014). Another commonly employed drying method is freeze-drying (FD), which involves freezing up the water followed by vaporizing (Chen and Wang, 2007). Due to decreased surface tension, FD keeps fiber morphology and porosity (Illa et al., 2019; Korhonen et al., 2011). Various benefits of drying hydrogels by these methods are given in Table 2.3.

The use of cellulose hydrogels has led us to think about the benefits of using gels derived from cellulose waste, which constitute an underutilized resource that can offer environmental as well as economic benefits (Behera and Mahanwar, 2020; Karunarathna et al., 2019).

2.5.1.(b) Synthetic hydrogels

Synthetic hydrogels are modified petrochemical-based modified polymers that contain functional groups like peptides, oligonucleotides, and cleavable linkages (Behera and Mahanwar, 2020). Water- soluble acrylate and acrylamide synthetic monomers are often employed for the synthesis of the cross-linked polymeric hydrogel.

To manufacture these hydrogels, the free radical copolymerization method is most commonly used, which involves reacting hydrophilic monomers with cross-linkers. To synthesize hydrogels for various applications, many synthetic monomers were copolymerized with acrylate- or acrylamide-based monomers. Hydrogels manufactured from poly (acrylamide-co-acrylic acid) have been used to hydrate soils in gardens and fields. Kim et al. (2010) demonstrated that commercially available polyacrylamide hydrogels modified with ionic groups have minimal influence on crop life and yield. However, the authors claimed that such synthetic hydrogels are unstable and performed poorly when it came to overall plant production (Simoni et al., 2017).

Table 2.3: Different hydrogel drying types with their advantages

Drying Type	Advantages
Freeze-drying (lyophilization)	Primarily used in the drying of cellulose crystals; maintains part of the fibres in nanoscale, though a relatively stable process.
Oven drying (solvent evaporation)	Low cost and quick process.
Spray drying	Continuous process, low operational cost, used mainly in drying nano-crystal pulp.
Supercritical drying	Good efficient drying, keeping the nanoscale of cellulose fibrils, used mainly for drying cellulose fibres.

2.5.2 Advantages of cellulose-based hydrogels

Cellulose hydrogels have improved biodegradability, better hydrophilicity, swelling-re-swelling activity, and biocompatibility as compared to traditional hydrogels that use acrylate or acrylamide as their monomers (Kabiri et al., 2011). Furthermore, due to their high absorptive capacities, these hydrogels act as a reservoir, storing excess water and nutrients in the agricultural field (Li and Chen, 2019). As a consequence, the demand for water and nutrients may be decreased and increased efficiencies may be achieved. Renewable cellulose-based hydrogels are more advantageous, in comparison to synthetic hydrogels. Synthetic hydrogels can be harmful to living organisms as they degrade from polyacrylamide to acrylamide. From this viewpoint, hydrogels derived from cellulose waste are profitable sources that can offer environmental as well as economic benefits (Simoni et al., 2017).

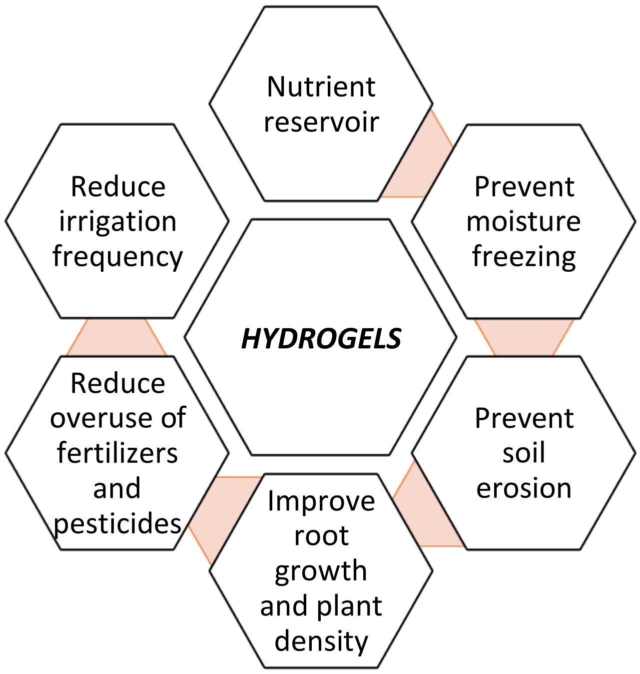

Figure 2.1: Applications of hydrogels in agriculture

Figure 2.1 depicts the numerous applications of hydrogels in agriculture (Singh et al., 2021). Hydrogels also have the advantage of being able to release water that has been absorbed into their surroundings, and they can be rehydrated with water once they have dried. Hydrogel polymers have been used to retain water in arid and semiarid environments where irrigation resources are limited or saline conditions hinder agricultural growth and yield (Singh et al., 2021). Apart from this, they are also used in drug delivery, solute separation, baby diapers, biomedicine, cosmetics, firefighting, biosensors, tissue engineering, food, printing inks, and textile application (Behera and Mahanwar, 2020).

2.6 Measurement of plant response to soil moisture and nutrients

With the increasing environmental and soil quality effects due to the over-application of water and fertilizers, soil tests have become important to assess the extent of surplus or deficit of nutrients if exists. Accordingly, precise measurements of soil moisture and macronutrients (i.e., N, P, and K) are required for efficient agricultural production (Kim et al., 2009). Various methods to measure moisture content range from laboratory methods to various in-situ techniques, while the measurement of crop nutrients range from laboratory methods, specific ion electrodes, colorimetric techniques, and other spectroscopy methods (Motsara, 2015).

2.7 Soil moisture content

Soil water (or moisture) content (SWC or θ) represents the relative amount of water present in the soil (Taşan and Demir, 2020). Most commonly, it is known as volumetric water content and expressed in percentage per volume. Theoretically, soil moisture content ranges from saturation (θ_{sat}) to the permanent wilting point (θ_{pwp}) (Novák and Hlaváčiková, 2019). The condition when soil is fully saturated is not considered ideal for plant growth and may decrease yield by depleting oxygen in the root zone.

Soil field capacity (θ_{fc}) is characterized as the amount of soil moisture after drainage of excess water (de Oliveira et al., 2015; Jabro et al., 2020), while the permanent wilting point refers to the point when the plant cannot access water, *i.e.*, the availability of water for the plant growth is zero (Nolz et al., 2016; Taşan and Demir, 2020). At θ_{pwp} plants start to wilt and do not recover upon wetting. The θ_{pwp} occurs when the soil matric potential (ψ_m) or pressure head reaches 1.5 MPa (i.e., $\psi_m = -1.5$ MPa), whereas the soil field capacity occurs when $\psi_m = -33$ kPa (Figure 2.3) (Jabro et al., 2020). The optimum growth condition for a plant is at θ_{fc}, yielding the most efficient crop production.

2.7.1 Plant available water content

Total plant-extractable water (TAW) is the quantity of water available for uptake by crop roots. It is defined as the amount of soil water between θ_{fc} and θ_{pwp} (Equation 1) in a field soil (Naggar et al., 2020). It is that portion of water in the soil profile, that can be easily extracted by a crop in a particular soil volume (Novák and Hlaváčiková, 2019). The equation for total available water content (TAW) can be expressed in mm, as mentioned in equation 1.

$$TAW \text{ (mm)} = \text{rooting depth} * (\theta_{fc} - \theta_{\text{pwp}}) \quad (1)$$

where,

TAW = Total available soil water in the root zone (mm)

θ_{fc} = Moisture content at field capacity in the soil volume(mm)

θ_{pwp} = Moisture content at the wilting point is considered soil volume (mm).

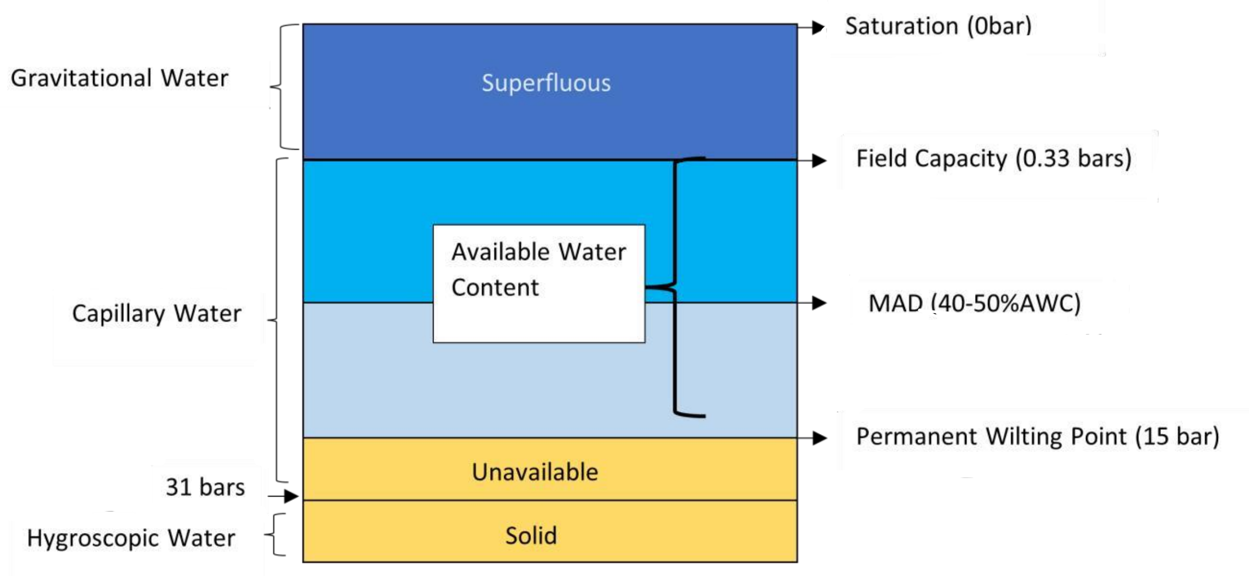

Figure 2.2: Soil water reservoir components

Figure 2.2 represents three different forms of water present in the soil (*i.e.*, gravitational water, capillary water, and hygroscopic water) with the soil water thresholds (*i.e.*, saturation, field capacity, permanent wilting point, and oven-dry condition (Hartge et al., 2016).

In a soil profile, the soil is considered saturated when all the pores (macropores and micropores) are occupied with water and the soil $\psi_{\text{m}} \approx 0$. This gravitational moisture is referred to as free moisture, where water moves quickly through soil macropores due to the force of gravity. As the water drains quickly, this moisture is considered as non-available moisture content. The soil micropores contain capillary water, and movement is through surface tension and capillarity. When the gravitational water is drained out, the remaining moisture can be defined as capillary moisture. They are retained and operated against the force of gravity with cohesive and adhesive forces. Hygroscopic moisture is the water retained by strong adhesion forces held very close to the soil particles and is not accessible to the plant (Lekshmi et al., 2014).

2.7.2 Soil moisture measurements

There are several ways to determine the soil moisture which can be categorized as laboratory methods and in-situ strategies. The direct strategies incorporate gravimetric soil moisture techniques. More automated techniques are prevalent, such as soil resistivity sensors, tensiometers (Mendes et al., 2019), and dielectric methods, such as time-domain reflectometry (TDR) (Ihuoma and Madramootoo, 2019, 2020), frequency domain reflectometry (FDR), capacitance techniques (Lim et al., 2017).

2.7.2.(a) Gravimetric method

The thermo-gravimetric technique is the standard reference in estimating the soil moisture content. Soil cores of approximately 100g are collected and the wet weight is determined. The cores are then set in the oven for at least 24 hours at 105 ºC. The soil moisture is estimated as the difference between the dry and wet weight. The significant disadvantage of the technique is that it is time-consuming, tedious, and requires destructive sampling, which disrupts the soil structure. Additionally, it cannot be applied in real-time irrigation scheduling as it requires 24 hours to calculate the soil moisture.

To enhance crop production and water use efficiency, accurate irrigation scheduling methods are required to reduce water loss and decrease the negative impact on the environment. Both under and over-irrigation have detrimental effects on the crop yield, increased runoff and nitrate leaching respectively. Thus, in assessing soil moisture content, the use of sensor probes is generally suggested instead of the gravimetric technique (Naggar et al., 2020).

2.7.2.(b) In-situ soil moisture sensing methods

Kashyap and Kumar (2021) referred to a non-destructive real-time point-based approach for field soil moisture measurement. These sensors can readily be calibrated to measure moisture content at different depths. As a result, farmers can use them in agricultural applications with ease (Kashyap and Kumar, 2021).

Moisture sensors, also known as capacitance resistance sensors, are one of the indirect methods for measuring soil moisture content by placing sensors at various depths in the soil. It determines the dielectric permittivity of the soil medium, which is then converted into soil water content. Decagons are equipped with a variety of sensors, including the EC-5;10HS; 5TM; 5TE; GS1, GS3, and Theta Probe sensors (Lim et al., 2017).

The Delta-T Theta probe (Ltd, 2016) sensor is an impedance sensor working at 100 MHz (Bretreger et al., 2020). For the quantification of soil moisture, knowledge of the dielectric constant is crucial. The radio signals produced by the probe, which are communicated through the hardened steel poles are used to determine soil moisture content. When embedded into the soil, a portion of the radio signal is dampened by the soil with the measurement of the reflected signal (Delta-T Devices Ltd., 2016). The ratio of the transmitted and received signal is converted to a voltage of between 0 and 1 Volts. Cited precision is ± 5%, using manufacturer calibration, within a soil moisture range of 5-50% (Bretreger et al., 2020).

Figure 2.3: Delta-T Theta Probe for measurement of moisture content (Delta-T Devices Ltd., 2016)

2.8 Measurement of crop nutrients

2.8.1 Laboratory methods for soil nutrient analysis

For the extraction and quantification of nutrients, sampling is done as per soil testing procedures (Barbanti et al., 1994; Brown, 1998; Morris et al., 2018). To begin with, extractant solutions are added to the soil sample and used to extract the macronutrients. The macronutrient-bearing extractant solution is then separated from the sample (usually by centrifugation), allowing for the determination of total available soil nutrient pools. Numerous nutrient extraction chemistries have been developed, the majority of which are pH dependent. These include N extraction using calcium sulfate or potassium chloride, P extraction using the Mehlich 3 method, and K extraction using the Mehlich 3 method (Brown, 1998).

Colorimetry is one of the commonly used soil testing methods in laboratories prior to the introduction of more advanced and sophisticated instruments. When a sample extractant reacts with a prescribed reagent, colour, or turbidity changes in response. This change is directly proportional to the concentration of a particular ion. Many colorimetric reagents have been developed to detect various soil nutrients such as NO_3^- detection via the diazotize dye method with the use of Cadmium; NH_4^+ detection via Nessler reagents, P detection via the stannous chloride method, K detection via tetraphenyl boron precipitation, and more. In order to determine concentration levels, the colour of the final product is compared to a reference colour strip (Dimkpa et al., 2017).

Compared to calorimetry, spectroscopy techniques enable more precise and rapid analysis while requiring less soil preparation. For laboratory-based soil testing, spectroscopic techniques such as visible (vis), ultraviolet (UV) (Fernandez et al., 2017), and infrared (IR) spectroscopy, X-ray fluorescence (XRF), and inductively coupled plasma spectroscopy have been commonly used. The interaction principle upon which these spectrophotometers operate is that photons of specific energy (or wavelength) are absorbed by the electron in orbitals and are observed in the absorbance spectrum. A chip-level colorimeter sensor based on Lambert's beer–equation was developed to identify nutrients in the soil (Kim et al., 2009; Liu et al., 2016). Ehsani et al., (1999) used near-infrared absorbance, Fast Fourier Transform, and a partial least squares regression to determine soil nitrogen in the range of 0 to 300 mg kg^{-1}.

However, soil monitoring using calorimetric and atomic emission spectroscopy based on manual or mechanical soil sampling is expensive, time-consuming, and applies to in-situ testing. Additionally, during transport and storage prior to testing, unwanted mineralization and nitrification/denitrification of the samples may occur (Ali et al., 2020).

Hence, advancements in the use of electrochemical sensing primarily based on ion-selective electrodes have been identified as beneficial in the real-time evaluation due to their ease to use, portability, quick response time, and ability to immediately test the analyte with a wide range of sensitivity.

2.8.2 Ion-selective membrane-based electrochemical methods

The majority of the electrochemical techniques are based on the use of an ion-selective electrode (ISE) or an ion-selective field-effect transistor (ISFET) to determine soil nutrient ranges. Both ISE and ISFET operate on the same theoretical principle, *i.e.*, they respond selectively to a specific ion in a solution (Barbanti et al., 1994), and their level can be assessed by a logarithmic correlation between ionic activity and electric potential. The ISEs and ISFETs require some essential elements, i.e., ion-selective membranes, that can be integrated with a reference electrode and convert the chemical response (ion concentration) to a signal (electric potential). ISE's have emerged in a variety of places throughout the world as a result of the growing demand for determining the number of recent ions, as well as great breakthroughs in the creation of numerous ISFETs. The most essential soil nutrients, such as NO_3^-, K, Na, Ca, Mg, and Cl, may be detected using ion-selective membranes, which are now commercially available (Kim et al., 2009).

Amongst the essential crop nutrients, soil N is absorbed by the plants in the form of NO_3^-, but in some acidic and/or anaerobic situations, NH_4^+ can be dominant. It is widely accepted that co-provision of NO_3^- and NH_4^+ may be optimum for growth of plant (Hachiya and Sakakibara, 2017). The inorganic form of nitrogen, $NO_3^- - N$ is prone to leaching while its constant supply is essential for plant growth and development. The optimal $NO_3^- - N$ concentration range in the soil is between 10 and 30 mg kg^{-1}, or 0.1 and 0.5 mM, which is within the detection range of the majority of the ISM-based detectors.

Ali et al., (2020) developed a deployable electrochemical soil $NO_3^- - N$ sensor by coating a printed circuit board with a nanocomposite of molybdenum disulfide (MoS_2) and poly (3-octyl-thiophene). To promote sustainable and economically viable agriculture, sensor technologies such as satellite imaging and data acquisition, as well as network wireless systems, are being employed. In soil slurries, this ion-selective membrane-based sensor can detect up to 1500 ppm of NO_3^--N. Moreover, low-cost soil $NO_3^- - N$ sensors have been developed by companies such as Horiba, Ltd., Hanna Instruments and YSI Inc. (Ali et al., 2020).

One of the widely used meters is the compact NO_3^- ion meter 'TwinNO_3', manufactured by Horiba (Kyoto, Japan) (Parks et al., 2012). The meter is pocket-sized with a display screen, a sensor pad containing electrodes and can measure in the range of (6-9900) ppm $NO_3^- - N$. The calibration of the 'TwinNO_3' for soil involves a two-step process, *i.e.*, calibration is performed with two different standard solutions of 150 and 2000 ppm, before dropping the soil sample onto the sensor. A voltage develops between the 2 electrodes, and the magnitude of this voltage reflects the extent of NO_3^- within the solution (Parks et al., 2012). Hence, this phenomenon allows us to estimate the NO_3^- concentration in the soil sample.

2.8.3 Biosensing techniques

Soil macronutrients such as N and P have been detected by biosensors in addition to ISM-based EC devices. Enzymatic biosensors, molecularly imprinted polymer (MIP), aptamers, and electro-catalysis-based detection methods are some of the most recent biosensing approaches (Kim et al., 2009; Liu et al., 2016).

2.9 Tomato response to water and nutrients

Tomatoes (Solanum lycopersicum Mill.) belong to the nightshade family Solanaceae and are one of the most frequently farmed and consumed vegetables in the world. After potato, it is the most valued vegetable crop and is well-known for its multiple health benefits: high antioxidant levels; low levels of fats and sodium (Cammarano et al., 2020; Madramootoo et al., 2021).

The crop is a daylength- sensitive warm-season crop that is susceptible to frost and freezing temperatures. Tomatoes can be grown in both open fields and greenhouses. The crop is high in vitamins, minerals, and antioxidants, all of which are necessary for a well-balanced human diet (Gebremariam, 2015). The daylength tomato plant has a growth period of 90 - 150 days. Various growth stages include the initial period, vegetative growth, flowering, and senescence period (Shamshiri et al., 2018). The ideal mean day-by-day temperature for viable growth is 18 to 25 ºC with a night temperature in the range of 10 and 20 ºC. The yield is susceptible to bigger contrasts in day and night temperatures. Though it can grow well in all soils, it develops better in drained light loamy soil with a pH of 5 to 7 (Tan, 1990).

2.9.1 Water and nutrient requirements of tomatoes

Similar to most vegetable production, tomato requires a considerable number of agronomic inputs. Irrigation water required for optimal crop growth ranges from 400mm to 600mm (Cammarano et al., 2020) while N fertilization needs vary during the growing season. Various stages of crop development have a heavy influence on irrigation water needs. Amongst the various stages of tomato development, the flowering and fruit set stages require a considerable amount of water. Moisture stress or inconsistent water delivery may result in the cracking of fruits, blossom end rot, small fruits, and thus lower marketable yields (Ihuoma and Madramootoo, 2020).

Early-season irrigation should only be used when absolutely essential, as it can cool soil and move fertilizer out of reach of plants in the vegetative growth stage. Watering can be done more often once plants are established and soil temperatures have warmed up, based on soil moisture monitoring or meteorological data using a water budget approach (Ihouma and Madramootoo, 2019).

The water requirements of a crop can be expressed on the basis of evapotranspiration (ET), which is the quantity of water transpired by the plant and evaporated from the soil surface (Jaria et al., 2013). ET is influenced by growth stage and crop cover, as well as air temperature and relative humidity, wind speed, and intensity of light. To optimise crop yield, irrigation or rainfall is necessary to restore ET (Tan, 1990).

Starting from transplanting, the total water requirements (ETm) of a tomato crop grown for a 3–4-month period are 400 to 600 mm. The crop factor (Kc) determines water requirements based on reference evapotranspiration (ETo) in mm period^{-1}, for various crop development stages; for the initial stage 0.4-0. 5 (10-15 days), the vegetative growth stage 0. 7-0.8 (20-30 days), the flowering stage 1.05-1.25 (30-40 days), the senescence stage 0.8-0.9 (30-40 days),and at harvest 0.6-0.65 (FAO, 2021).

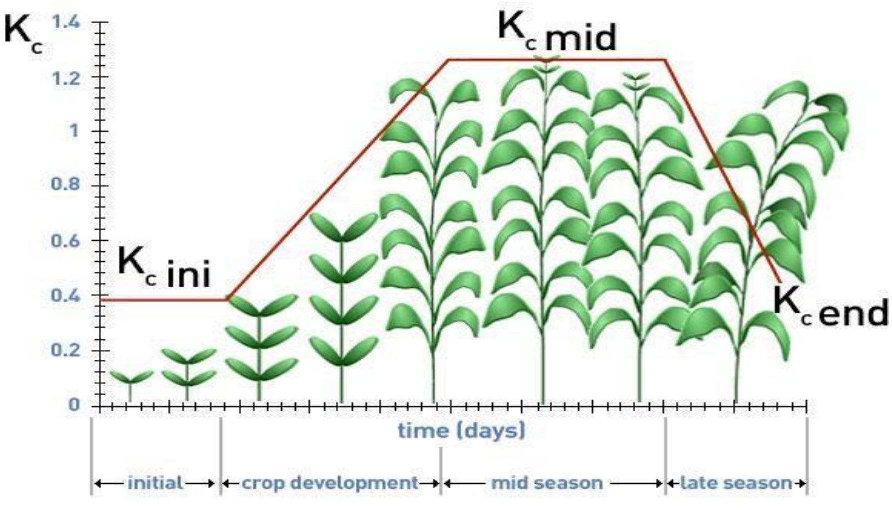

Figure 2.4: Crop coefficients at various stages of tomato crop growth (FAO, 2019)

The highest nitrogen demand occurs during vegetative growth with an overall uptake of 300 kg N ha^{-1} (Cammarano et al., 2020). According to FAO (2002), fertilizer requirements fluctuate between 100 to 150 kg N ha^{-1}, 65 to 110 kg P ha^{-1}, and 160 to 240 kg K ha^{-1}. As N management affects plant development and photosynthesis, N is usually applied in small doses, thereby preventing N losses that may arise due to immobilization or volatilization (Cammarano et al., 2020).

2.10 Literature review summary

This literature review presented an overview of the effective use of water and fertilizers through the use of hydrogels. The review began with a discussion of crop water and nutrient stress, followed by an innovative approach to deal with water and nutrient pressures. Hydrogels are of great scientific interest as they possess the capacity to retain and release a significant amount of water and nutrients when needed. Cellulose-based hydrogels are preferred over synthetic hydrogels, as they pose no risk to food safety or soil microbe populations and are ecofriendly. The importance of critical crop nutrients (N, P, K), their impact on plant growth, and different methods of nutrient measurement have also been discussed. Based on this literature review, there is a need to investigate the applicability of different hydrogels based on their drying methods, for crop growth. Specifically, there is a need to study freeze-dried and oven-dried hydrogels on crop yield, other crop physiological parameters, water and $NO_3^- - N$ use efficiency.

Chapter 3 : Materials and Methods

3.1 Experimental site

The study was conducted in the Plant Science Research Greenhouse at the Macdonald campus of McGill University, Ste. Anne de Bellevue, Canada (lat. 45° 26' 17" N, long. 73° 56' 17" W with an elevation of 36 m AMSL). The average growing season daytime temperature was 25°C, the night-time temperature was 16°C, and relative humidity 65%. The data collected was constrained to the specific soil type, crop (tomato), and climate of the greenhouse.

3.2 Preparation of hydrogels

The manufacturing of hydrogels from the office wastepaper was done by the dissolution, regeneration, and then finally the loading of the beads with 20-20-20 fertilizer according to the procedure of Gong et al. (2014). The steps followed in the preparation of freeze-dried and oven-dried hydrogels were as follows.

At first, the wastepaper was shredded using an electric spice and coffee grinder (KRUPS - Model No.- F20342) equipped with stainless steel blades and a capacity of 85.05g. The shredded paper was hydrolyzed in 15 wt% H_2SO_4 (aq), for 24 hours. To fabricate one batch of beads, 130 g of shredded paper (SP) was placed in 1000 mL of 15 wt% H_2SO_4 (aq), and the suspension was stirred at room temperature. The primary reason for pre-treating the paper by acid hydrolysis was, to dissolve cellulose to molecular weights lower than 1.0×10^5 g using a NaOH/urea aqueous solvent (Gong et al., 2014). Following the process of hydrolysis, vacuum filtration of the 130 g hydrolyzed paper (HP) was carried out, and after 24 hours, the HP was washed using distilled water, until the pH reached a neutral level (light green color on pH paper). In the next step, 130 g of the neutral HP was oven dried for 24 hours at 50°C. After collecting dried paper from the oven, it was ground once more to perform the further steps.

At the next stage of manufacturing, two distinct solutions were prepared to obtain the hydrogel beads. These solutions were comprised of 7 g of NaOH and 12 g of Urea (CH_4N_2O,), respectively, dissolved in 81 mL water. After that, 4 g HP was added to 96 mL NaOH/urea aqueous solution and mixed with the help of an IKA homogenizer (T10 basic ULTRA-TURRAX) operating for two minutes at 1000 rpm. The slurry formed with HP and the NaOH/Urea solution was frozen overnight at -20 °C, thawed to -12.6 °C and agitated at 2000 rpm for 15 minutes with an IKA homogenizer to generate a semi-viscous liquid solution. Drop-wise syringe extrusion in a 10 wt% H_2SO_4 aqueous coagulation bath at 0-4 °C was used to convert the cellulose solution into hydrogel beads. After 10 minutes in the liquid coagulation bath, which is primarily responsible for the phase inversion process for polymer precipitation, the beads were filtered and rinsed with distilled water until they reached a neutral pH of 7. The pH was measured using litmus paper. All of the beads were immersed in a commercial 20-20-20 N- P-K fertilizer (CF) solution containing 10% (w/v) for 24h. The fertilizer-loaded cellulose beads were then separated and dried using one of two methods: 1) oven drying (OD) and 2) freeze- drying (FD). For OD beads, the beads were dried in a standard oven at 50°C for roughly 4 days. In contrast, for the FD beads, the fertilized hydrogel beads were kept in a 12 L capacity LABCONCO Freeze dryer (S.No.-181166951), at a temperature of -50°C for 2 days.

The pictorial view of the process is illustrated in Figure 3.1 where the disintegration of the waste office paper (OP) and regenerated cellulose hydrogel beads with or without loaded commercial 20-20-20 fertilizer is shown (Gong et al., 2014).

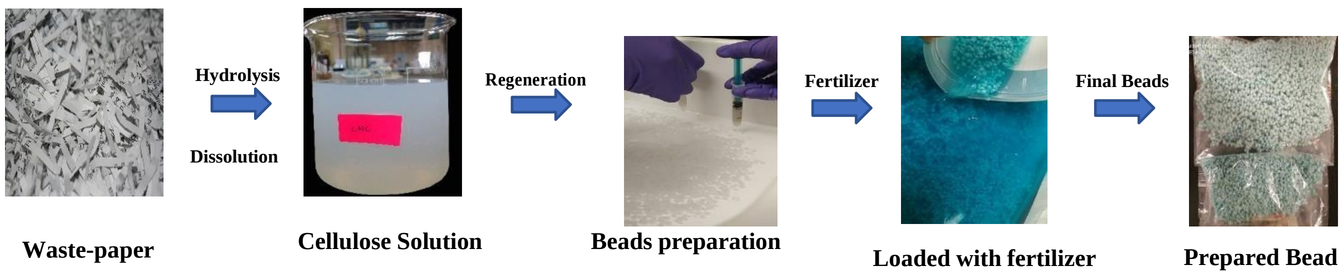

Figure 3.1 Hydrogel preparation from waste office paper

3.3 Experimental design

The experiment was performed in two phases, with and without a tomato crop: the first phase was for four months from February 15th to June 15th, 2021, and the second phase was from April 20th to May 24th, 2021. The hydrogels (freeze-dried, oven-dried) in each pot were incorporated with soil and compared with a control (contained fertilizer but no hydrogel). The study was conducted to assess the performance of two hydrogel treatments (HT) (freeze-dried and oven-dried) and a hydrogel-free control treatment i.e., a total of three treatments. These hydrogel treatments were coupled with two irrigation treatments (75% AWC and 95% AWC) using a 3 X 2 factorial design. Randomly arranged, the pots for both of the phases were placed on two benches, with a spacing of 0.6m X 0.6m between the pots. Freeze-dried (FDH), and oven dried (ODH) hydrogels beads were mixed in the soil in the middle of the pots, to a depth of 0.15 m from the top of the pots.

An important consideration was that the same 20-20-20 N-P-K fertilizer was used in the loading of the hydrogel beads and the control samples. Moreover, due to the different morphology of FD beads, OD beads and the third with regular fertilizer formulation, they all don't weigh the same. Therefore, it was impossible to incorporate the same weight of beads in all of the three treatments. Calculations were therefore done to determine the required number of beads needed to incorporate in the soil profile, taking into consideration that they have the same amount of fertilizer in all of the three treatments (Table 3.1). According to Oliva (2020), the optimum amount of fertilizer retained by FDH and ODH were 143.30 mg g^{-1} and 127.90 mg g^{-1}, respectively. Therefore, the necessary amounts, corresponding to 4.6 g of commercial fertilizer, were 35.96 g of FDH, 32.10g of ODH and 4.6 g of commercial fertilizer (Oliva, 2020).

Table 3.1: Quantity of hydrogel beads incorporated into the soil

Type of beads	Fertilizer required (g pot^{-1})	Fertilizer in beads (mg g^{-1} of bead)	g of bead per pot (g)
Freeze-dried beads	4.60	143.30	32.10
Oven-dried beads	4.60	127.90	35.96

3.4 Water application under various treatments

Based on available water content (AWC), which is the difference between the field capacity (FC) and the permanent wilting point (PWP), the various irrigation applications were scheduled. The upper irrigation thresholds were set at 95 % and 75% AWC for both AWC treatments, while the lower irrigation threshold was set at 10% manageable allowable depletion (MAD) for all hydrogel treatment combinations. Application of irrigation was initiated when the soil moisture in each pot was depleted beyond this MAD range, i.e., 85% for 95 % AWC and 65% for 75% AWC treatments and was terminated when the upper threshold i.e.,95% and 75% was attained. Based on the theta probe soil moisture readings, water was applied to maintain the pots at 95% AWC (39.85% m.c) and 75% AWC (35.25% m.c) levels, as shown in table 3.2.

Table 3.2 Moisture content calculations corresponding to upper and lower thresholds

AWC levels	Irrigation thresholds (AWC)	Moisture content (m.c)(%)
AWC – 1	Upper threshold – 95%	39.85
	Lower threshold – 85%	37.55
AWC – 2	Upper threshold – 75%	35.25
	Lower threshold – 65%	32.95

The main reason to include two different watering applications was to assess the performance of the crops under somewhat drought-like conditions versus no-stress conditions. The amount of water applied to each pot during irrigation was determined as the product of the Manageable Allowable Depletion (MAD), Available water content (AWC) and the rooting depth (RD) per pot, as adopted in Ihuoma and Madramootoo (2019). Irrigation was provided equally to all treatments at the beginning of transplanting, based on 100% replenishment of water in the plant root zone to field capacity for plants to establish; after that, the various irrigation treatments were introduced until harvest (Ihuoma, 2020).

3.5 Experiment based on hydrogel and water applications

3.5.1 Experiment conducted with tomato crop (Phase one)

Tomato (Solanum Lycopersicum 'Roma') seedlings were transplanted on 15th February 2021 into 18 pots (each 0.27 m wide * 0.32 m depth), with one seedling per pot, filled with mineral soil from the Horticultural Farm of McGill University. As per the experimental design, 6 hydrogel – AWC treatment combinations were replicated three times, giving a total of 18 experimental units (Figure 3.2).

Figure 3.2: Layout of pots in the greenhouse

This factorial design allowed examining interaction effects. The experimental layout is shown in Figure 3.3. Moreover, a water tap was connected to the bottom rim of the pots to drain surplus water, in order to maintain the required level of AWC in the pots.

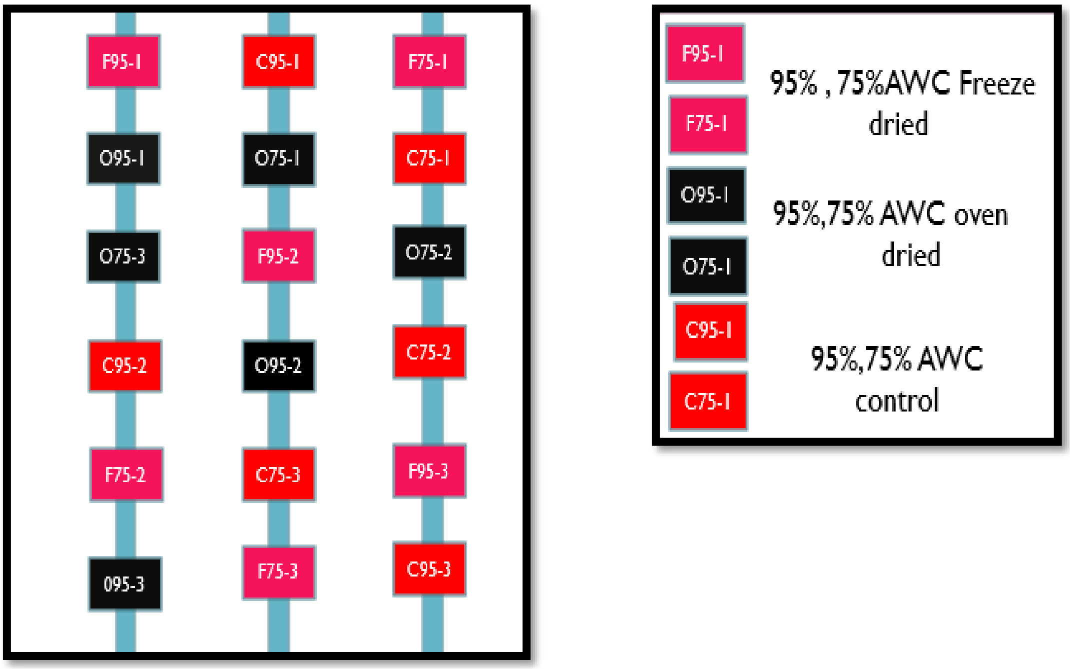

Figure 3.3: Factorial design layout used in the greenhouse study

(Where F, O, C signifies freeze dried hydrogels, oven dried hydrogels and a hydrogel-free control treatment with plant watering applications up to 75 % or 95% AWC; 1,2,3 represents the 3 replicates).

3.5.2 Experiment conducted without tomato crop (Phase two)

Phase two of the experiment was carried out without the crop to compare the nitrate release trends with and without the crop. The experiment was carried out from 20th April to 24th May under laboratory conditions, following the same factorial design (as explained in experimental design). This experiment also consisted of two HT (freeze-dried and oven-dried) plus the hydrogel-free control, *i.e.*, a total of three treatments, with two irrigation treatments (75% AWC and 95% AWC) i.e., a total of 3*2= 6 combinations, replicated twice, for a total of 12 experimental units.

3.6 Data collection

3.6.1 Soil texture of the soil

The hydrometer method, commonly known as the sedimentation method was used to calculate the particle-size distribution (PSD) of the soil used in the study. The hydrometer is a device used to measure changes in the specific gravity of the liquids over time and is usually expressed as the density of the liquid to that of water (Brockhaus and Carlozzo, 2012). The procedure was followed according to the ASTM standards, consisting of mixing 60 g of oven-dried soil in 125 ml of a 4 % solution of a dispersing agent (sodium hexametaphosphate- $Na_6[(PO_3)_6]$). All particles were in suspension when the soil was mixed. The test began at t = 0, but in one-minute, heavy particles tend to settle down at the bottom while silt and clay (very fine particles) remain in suspension. The calibrated hydrometers were used to obtain readings after 1 min, 2 mins, 5 mins, 15mins, 30 mins, 1 hour, 4 hours, 8 hours, 12 hours, and 24 hours. The texture of the soil was found to be in the class of silty clay loam. The key parameters, FC and PWP were determined, on the basis of soil texture and the bulk density of the soil. The percentage of sand (14%), silt (66%), clay (20%) and bulk density (1.2), were input into the RETC software (Koslowski et al.,2022; Villagra et al., 2021), the required FC (41%) and PWP (18%) were obtained from the model (Table 3.3).

Table 3.3: Texture and moisture retention of the experimental soil

%Sand	%Silt	%Clay	Bulk density (g cm^{-3})	θ_{fc} (%)	θ_{pwp} (%)
14	66	20	1.2	41	18

3.6.2 Soil moisture

Moisture content was measured daily using a Theta Probe sensor. Replenishment of water in the soil profile under the two different water applications 75% and 95% AWC) treated with the two different hydrogel treatments, and control was monitored using the Theta Probe sensor (Figure 3.4) to ensure that the daily irrigation requirements were met.

Figure 3.4: Measuring soil moisture content with Theta Probe

3.6.3 NO_3^- content

The NO_3^- content values were measured using a Horiba hand-held NO_3^- sensor (Model No.- UZ-05760-05), from GENEQ inc. Instruments Scientific (Ali et al., 2019). The sensor was advantageous due to its simplicity, ease, rapid response, and ability to directly measure the analyte with sensitivity. This provided a non-destructive and direct measure to calculate NO_3^- content with high precision. Each week, the NO_3^- content in all 18 pots was measured.

The process of measuring NO_3^- content involved extracting 5g of soil from the pots, followed by making a soil solution with a sodium sulfate solution, in a proportion of 1: 10 i.e., 5 g of soil with 50 g of sodium sulfate solution. The final step involved filtering the slurry through a Q5 filter paper. The resulting filtrate was then inserted into the sensor, and the soil NO_3^- content was obtained in ppm (Figure 3.5).

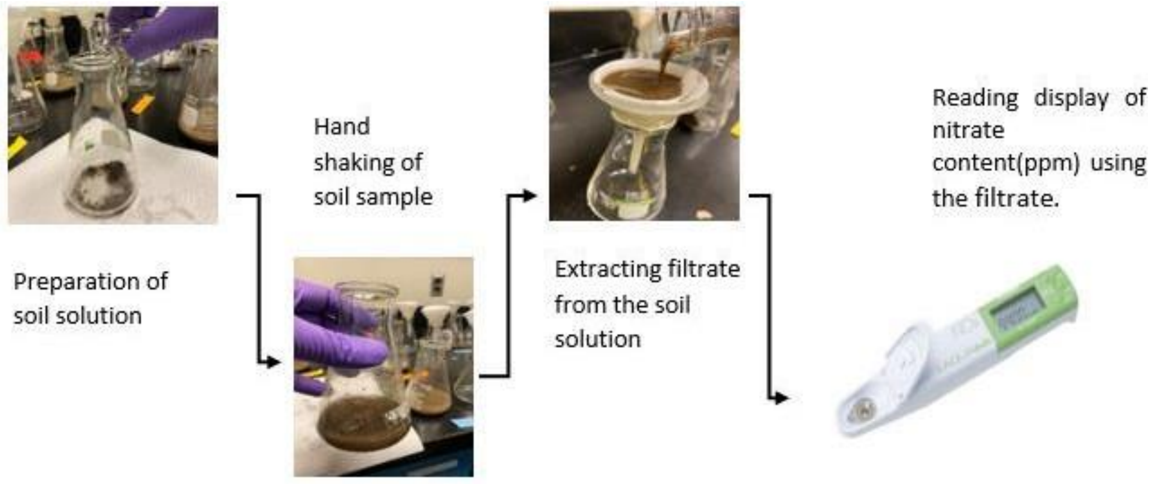

Figure 3.5: Procedure followed to determine the NO_3^--N *content*

3.6.4 Crop Parameters

Table 3.4 shows the physiological parameters of the tomato crop assessed along with the measuring device and measurement frequency.

Table 3.4: Crop physiological parameters with their measurement

Crop parameters	**Measuring devices**	**Frequency (weeks)**
Crop yield	Sorting marketable fruit, weight using lab scale	At fruit ripening
Plant height	Measuring Tape	Every three weeks
Plant weight	Lab-scale	Every three weeks
Stem diameter	Vernier calliper	Every three weeks
Number of fruits	Manual	Every two weeks
Number of flowers	Manual	Every two weeks

3.6.4.(a) Measurement of crop yield

Crop yield is defined as the weight of all harvested fruits from each plant sample per treatment. To avoid any errors, the number of fruits was always counted three times. The fruits were initially weighed and categorized as marketable or not, and their weights were totalled. Marketable fruits were those fruits with the highest consumer acceptability on the basis of size, shape, colour, as adopted in Ihuoma and Madramootoo (2019). While the total yield was measured as the sum of marketable and non- marketable fruits.

3.6.4.(b) Plant biomass

At the end of the harvest, the total biomass, including both roots and shoots of the plants was measured. The root system was separated from the remainder of the plant, and the rest was considered to be the total above-ground biomass, including any remaining non-marketable yield (Scholberg et al., 2000).

3.6.4.(c) Leaf area index

At the end of the harvest stage of the tomato growth cycle, three leaves, having the longest length and width, were plucked from each plant treatment^{-1}, sealed in an airtight container, and transported to the water innovation laboratory in the Macdonald Stewart building of McGill University. The samples were placed on the LICOR 3100 C Leaf area meter, operating with the specifications listed in Table 3.5. The LAI meter was calibrated, and various readings for different thresholds were recorded by placing the leaf on the middle section of the belt.

Table 3.5: Specifications of leaf area index meter

Resolution	**Display**	**Conveyor Belt speed**	**Light Source**	**Power Requirements**	**Operating Temperature**
1 mm^2 or 0.1 mm^2 (adjustable)	Full 8-digit LED	80 cm s^{-1} at 60 Hz; 6.7 cm s^{-1} at 50 Hz	15W fluorescent tube	108-126/216-252VAC, 48-46Hz, 100W max	+15- 55 °C

3.6.5 Water Use Efficiency

Water use efficiency (WUE) refers to the ratio of the water used in the plant metabolism to the water lost through transpiration. In this study, WUE was measured by calculating the total weight of fruits (kg) and the total amount of water applied to the plants (mm) (Lim et al., 2017; Bacon, 2009).

$$WUE = \frac{Marketable\ yield\ of\ the\ tomatoes(kg)}{Amount\ of\ water\ applied\ to\ the\ crop(mm)} \tag{2}$$

3.7 Statistical analysis

The statistical analysis involves a factorial design, with three treatments (FDH, ODH, and control) factorially combined with two irrigation thresholds (95% and 75% AWC), were conducted with SAS software (9.4 version, SAS Institute, Inc., Cary, NC, USA). The F-tests conducted by SAS PROC GLM (General linear model), determined whether or not, treatment has an effect on the plant height, stem diameter, total marketable yield, leaf area index, and plant biomass and irrigation water use efficiency. To test for the significance between various treatments, adjustments for multiple comparisons were made using Bonferroni’s method, as suggested by Mendes et al. (2019).

Chapter 4 : Results and discussions

4.1 Estimation of moisture content

4.1.1 Calibration curve of Theta Probe moisture sensor

The measured Theta Probe readings were calibrated by gravimetric soil moisture measurements to obtain a calibration curve (Figure 4.1). A wide range of moisture contents were used to calibrate the theta probe soil moisture sensor. The water was applied to the test pots in the range from 0.76 – 0.6 $cm^3\ cm^{-3}$. This was strictly for calibration purpose and are not reflective of the moisture contents during the greenhouse experiments with a crop. The curve helped in estimating the actual values of the daily moisture content.

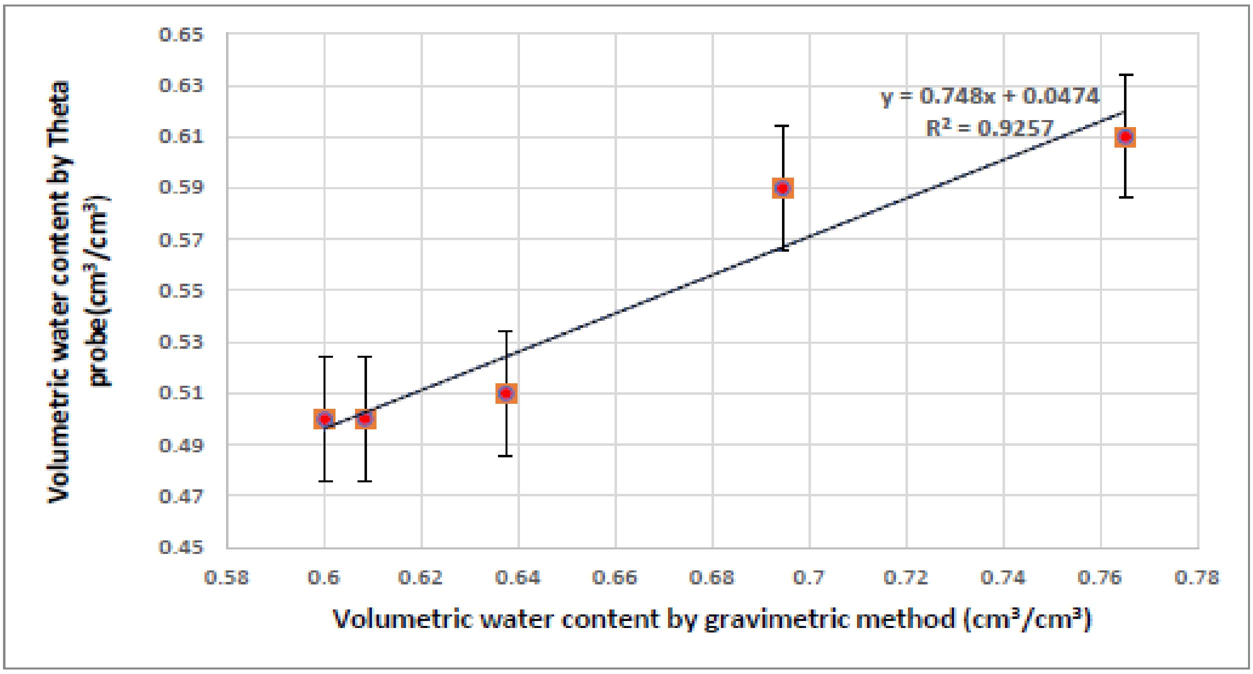

Figure 4.1: Calibration curve for theta probe moisture sensor

4.1.2 Water applied under different treatments

Volumetric soil water content (SWC) for the two different irrigation regimes incorporated with hydrogel treatments are represented in Figure. 4.2. Various water treatments (75% and 95% AWC) showed that SWC was low in 75% AWC ranging from 30 to 35% while 95% AWC was kept at a higher range between 37 to 42%.

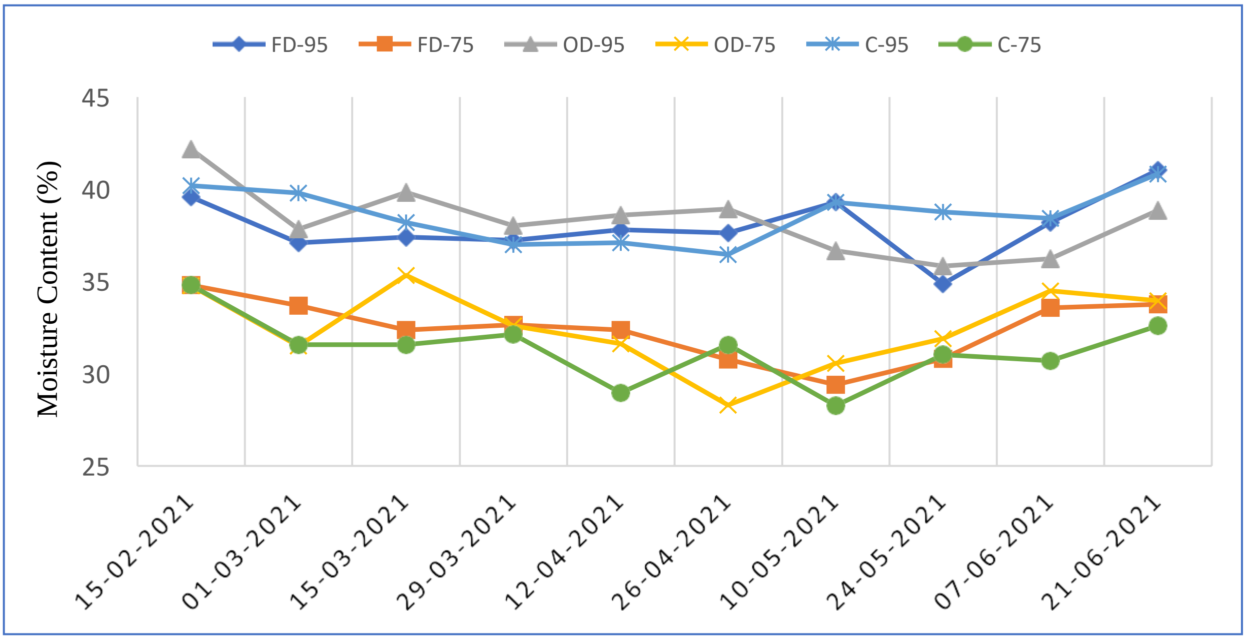

Figure 4.2: Variation of soil moisture during the growing season.

The total water applied for the 95% and 75% irrigation thresholds ranged from 225 to 265 mm and 178 to 195 mm, respectively, during the entire growth period. For both thresholds, the control, which did not contain hydrogels, used the maximum amount of water (265 mm for 95% AWC and 195 mm for 75% AWC), followed by FD and OD hydrogels, as shown in Table 4.1.

Table 4.1: Total water applied (mm) for tomato plants per growth stage for each treatment

		Treatments (% of AWC)					
Growth stages	**Days**	**F-95%**	**O-95%**	**C-95%**	**F-75%**	**O-75%**	**C-75%**
Initial	30	32	35	38	21	25	29
Vegetative growth	40	63	59	77	56	51	58
Flowering	45	79	72	93	61	59	66
Maturity	20	51	52	57	40	38	42
Total	135 days	225 mm	218 mm	265 mm	178 mm	173 mm	195 mm

From Table 4.1, it is evident that controlled treatments require more moisture during the growth and development stage compared to the freeze and oven dried hydrogel treatments. The findings of the study are in line with the findings of Pereira et al. (2017), where chitosan-based natural hydrogels absorb and steadily swell in distilled water. As a result of their absorption, water molecules take up the space of their three-dimensional network and hydrolyze the polymeric urea material in the polymer chain. Thus, the hydrolyzed urea holds water inside the 3-dimensional network and releases it gradually to the plant through the process of dynamic water exchange. A study carried out by Rehim et al. (2004) revealed that the seeds around the hydrogel can extract a large amount of water from the hydrogel formulations. In their study, there was an increase in early seed germination in hydrogel-treated samples as compared to the control, which are without hydrogels. Figures 4.3 and 4.4 show that the control in both of the AWC levels has a higher demand for water in all of the crop growth stages, as compared to other treatments, making hydrogels more useful in arid and semi-arid regions for agricultural water management.

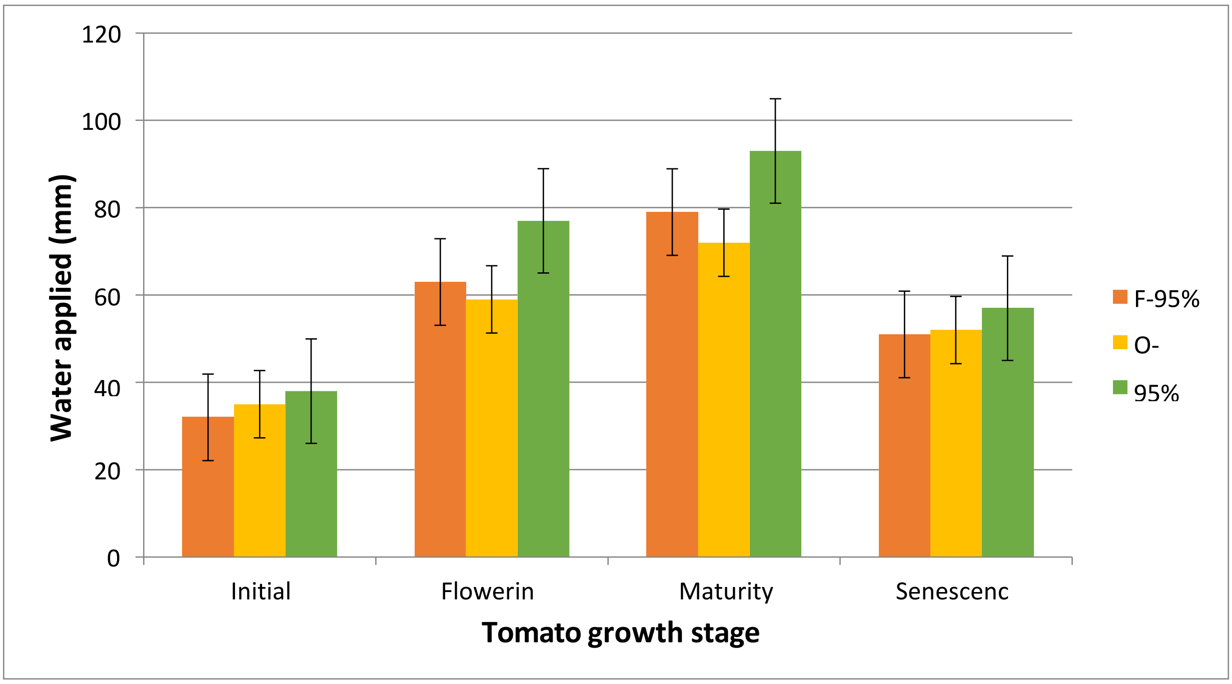

Figure 4.3: Water applied (mm) to hydrogel treatments with 95% AWC

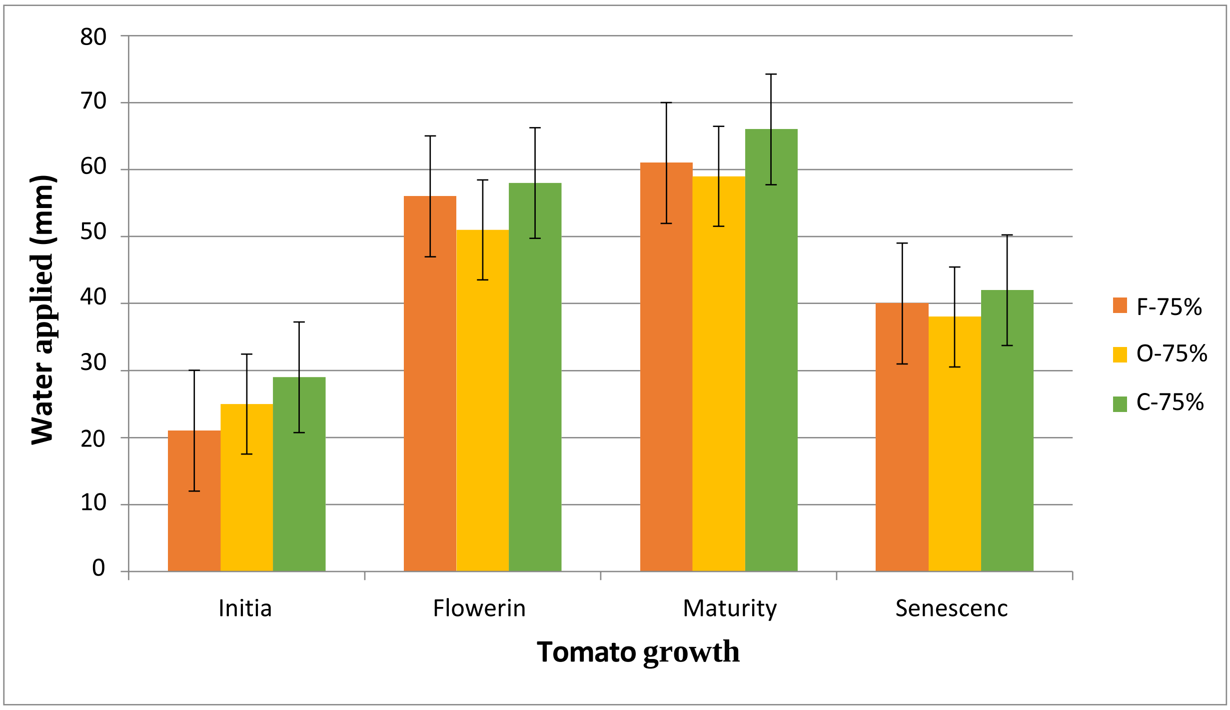

Figure 4.4: Water applied (mm) to hydrogel treatments with 75% AWC

4.2 Estimation of NO_3^- – N content

4.2.1 Calibration curve of NO_3^- – N content

The NO_3^- – N sensor was calibrated with the help of standard solutions available for different NO_3^- contents (ppm), and the corresponding graph was obtained (Figure 4.5), which was used to measure the NO_3^- – N content in the pots at every phase of the growing season. Generally, the Horiba sensor gives a higher nitrate content reading. Therefore, it is recommended to be calibrated against laboratory standards. These calibrations resulted in a coefficient of determination (R^2 = 0.9818) close to 1. Hence the calibration equation for was used to compute the actual nitrate content.

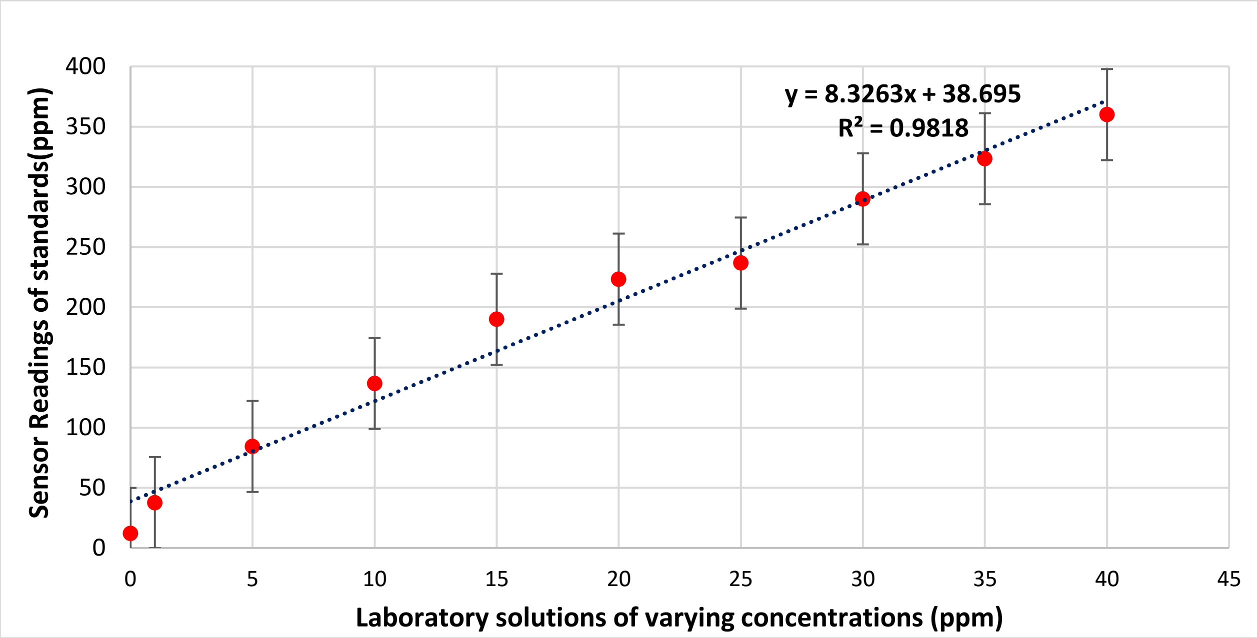

Figure 4.5: Calibration curve for nitrate content sensor

4.2.2 NO_3^- - N release of hydrogels without tomato crop.

Figure 4.6 demonstrates the results obtained, between the concentration of nitrogen in the form of $NO_3^- - N$ and various treatments (FDH, ODH, Control). Amongst the 75% AWC thresholds, the $NO_3^- - N$ concentration of FDH-75 treatments increased from 14.53 to 37.68 mg kg^{-1}, followed by a slight decrease in the concentration to 25.21 mg kg^{-1}. While for 95% AWC, the results showed a three-fold increase in FDH-95 from 21.30 to 66.53 mg kg^{-1}during a month.

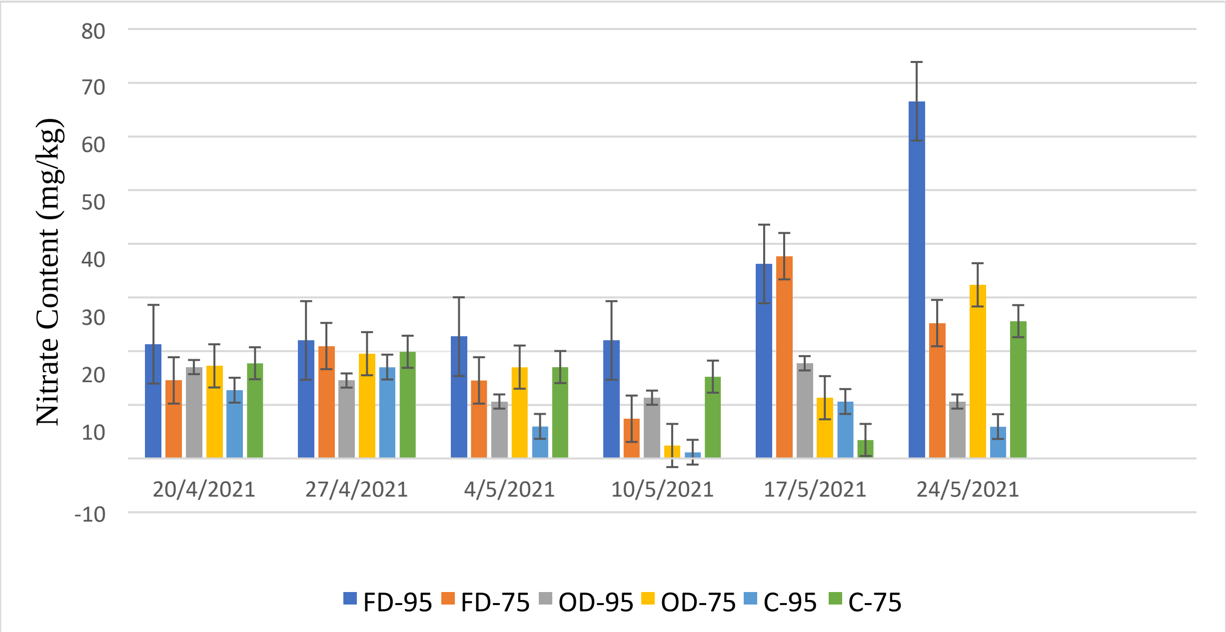

Figure 4.6: $NO_3^- - N$ content release curve without crop

On the other side, $NO_3^- - N$ curves for ODH-95 and Control-95 treatment showed that these treatments have a minimum release, with the values being consistent in a range of 10- 20 mg kg^{-1} during the entire phase of the experiment.

The investigation carried out by the study shows that the FDH treatments effectively release the fertilizer from the hydrogels to the plants, compared to the other treatments. This result is in the line with the findings of Abobatta (2018), where the slow-release hydrogel polymer served as a primary approach to retain the nutrients and release them gradually throughout the growth cycle. Moreover, a study conducted without a crop that does not form storage organs showed a similar pattern of NO_3^--N deposition, with NO_3^--N typically continuing to accumulate with the developing plant's age (Anjana et al., 2006), as shown in Figure 4.6 for FDH treatments. The results of FDH-95 demonstrated that the hydrogel formulations with freeze- drying prohibits nutrient leaching by decreasing runoff and controls the fertilizer release over a long period of plant growth (Ni et al., 2009).

4.2.3 $NO_3^- - N$ content during the tomato growth phase

Figure 4.7 and 4.8 demonstrates the release of $NO_3^- - N$ content (mg kg^{-1}) during the 4-month tomato-growth phase. During the initial growth stage of the tomato crop, FDH treatments exhibited consistent values ranging from 17 to 19.5 mg kg^{-1} (Figure 4.7) for 95 % AWC. When the vegetative development stage ends, the concentration of $NO_3^- - N$ in the soil dropped to 4.9 mg kg^{-1} and a continuous decline was observed till the end of the senescence stage (1.1 mg kg^{-1}) (Figure 4.8). This signifies that the fertilizer concentration in the form of $NO_3^- - N$ was gradually increased until the start of the vegetative development stage, and the increased $NO_3^- - N$ was gradually released and absorbed by the tomato crop, in subsequent stages. The imbalance in $NO_3^- - N$ concentration is caused by net absorption and assimilation rates, which is a relatively photosynthetic mechanism that occurs in many plants (Ferrario-Mery et al., 1997).

While for the control treatments, which were without hydrogels, the continuous decreasing trend was observed throughout the growth cycle, from 6.1 mg kg^{-1} to 2.1 mg kg^{-1}. The control plants indicated a lower value at the start of the growing season, which suggests that the control treatments were unable to provide adequate nutrients to the plants.

ODH showed a slightly different pattern than the other two treatments, in terms of consistency. Initial $NO_3^- - N$ concentration after inserting the hydrogels was found to be 5.8 mg kg^{-1}, which tripled to 18.2 mg kg^{-1} (Figure 4.8) during the end of the flowering growth stage, with a gradual release in the concentration of $NO_3^- - N$ content to 1.5 mg kg^{-1} at the end of the season. The findings of the research show that the excess of the $NO_3^- - N$ was taken up by the crops (from initial to flowering stage) and was most likely stored in the vacuole where it can be remobilized when the supply of nitrogen is inadequate (senescence stage), so as to meet the demands of the crops.

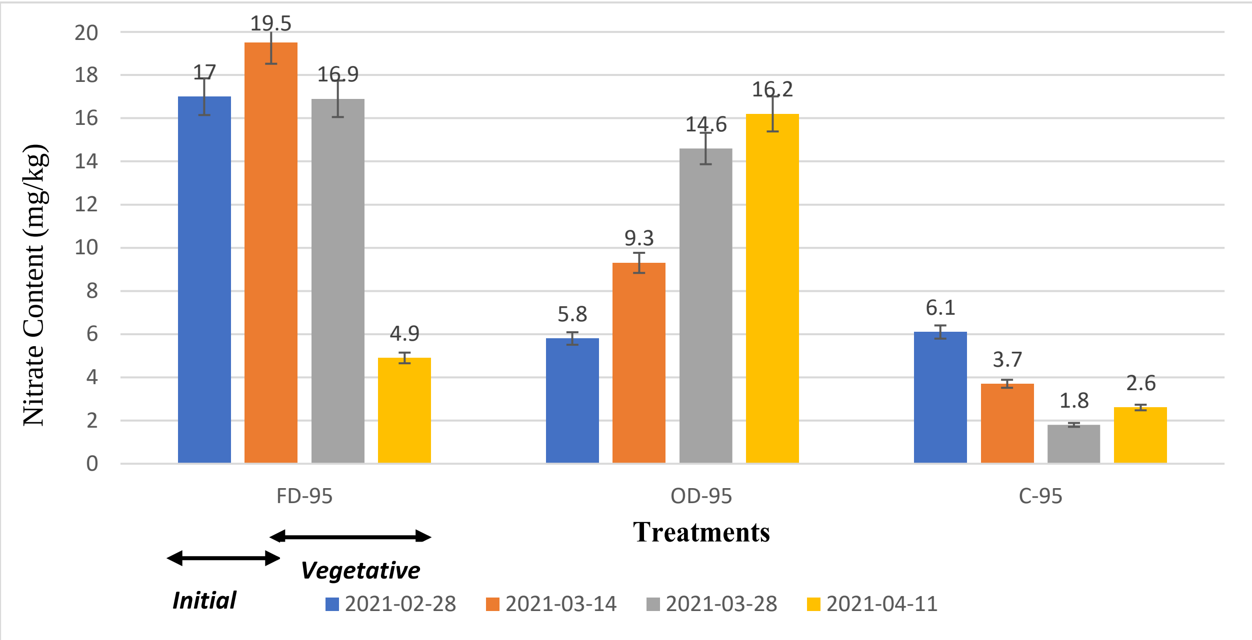

Figure 4.7: $NO_3^- - N$ content release curves from initial to vegetative growth stage with 95%AWC

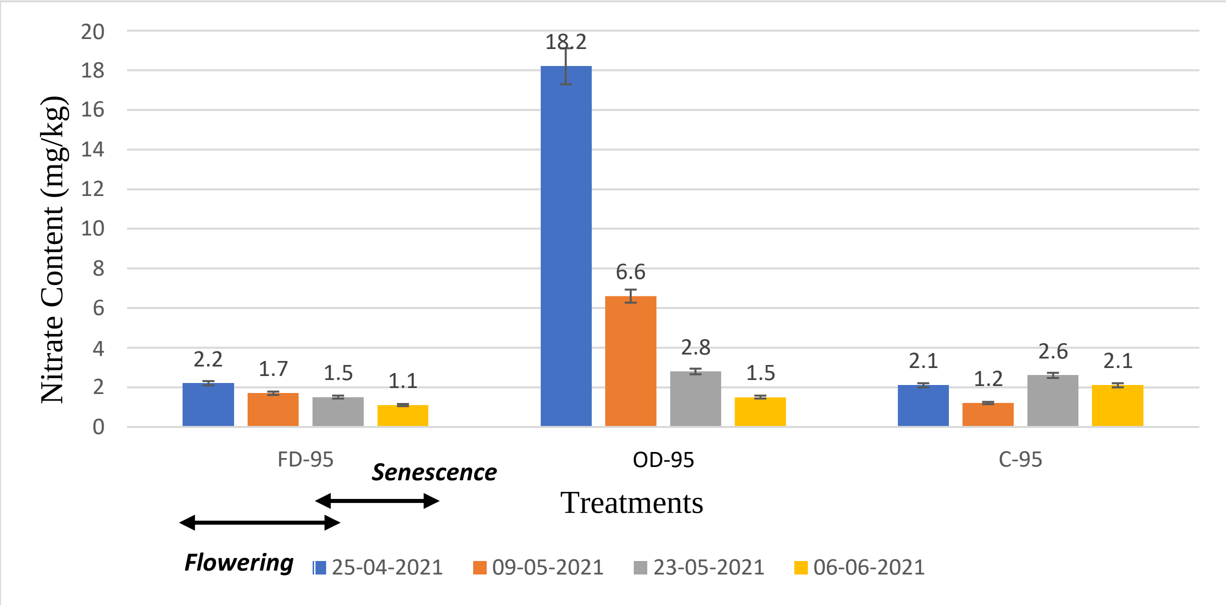

Figure 4.8: $NO_3^- - N$ content release curves from flowering to senescence growth stage with 95% AWC

For 75% AWC (Figure 4.9 and Figure 4.10), FDH treatments showed a consistent decreasing pattern throughout the growth period, with a decline from 28.6 mg kg^{-1} to 1.1 mg kg^{-1}. While ODH and the control showed a slightly different pattern, in comparison to FDH, maintaining consistency throughout the growing season. Initial $NO_3^- - N$ concentrations for control and ODH treatments were found to be 9.8 and 7.7 mg kg^{-1}, following a decline in the amount of $NO_3^- - N$ content to 1.4 and 2

mg kg^{-1} at the end of the season.

The low concentrations of the $NO_3^- - N$ in ODH and control treatments, in comparison to FDH, during the starting of the crop growth season, suggests that nitrogen availability was limited in the crops (McCall and Willumsen, 1999). In order to avoid these limiting $NO_3^- - N$ at the starting of the growth season, biweekly application of fertilizer was provided. The rise to 11.3 mg kg^{-1} recommends that the plant start to accumulate the $NO_3^- - N$ with the increase in nitrogen fertilization (Chen et al., 2004). Moreover, lower values of $NO_3^- - N$ are found during the last stage of the crop growth cycle in all the treatment combinations, which is consistent with the findings of Anjanan and Iqbal (2007), which shows that the plants that develop fruit, primarily potato and tomato, have lower $NO_3^- - N$ concentrations in the petioles, during the harvest. The lower value in the petiole is mostly determined by the growing storage organ or the translocation of soluble nitrogen.

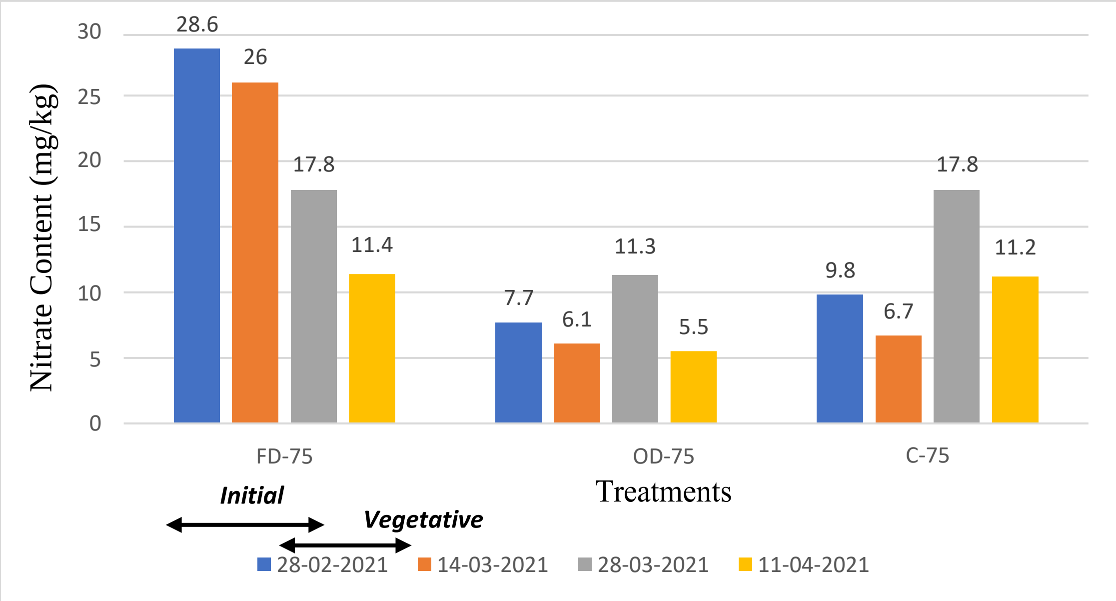

Figure 4.9: $NO_3^- - N$ content release curves from initial to vegetative growth stage with 75% AWC

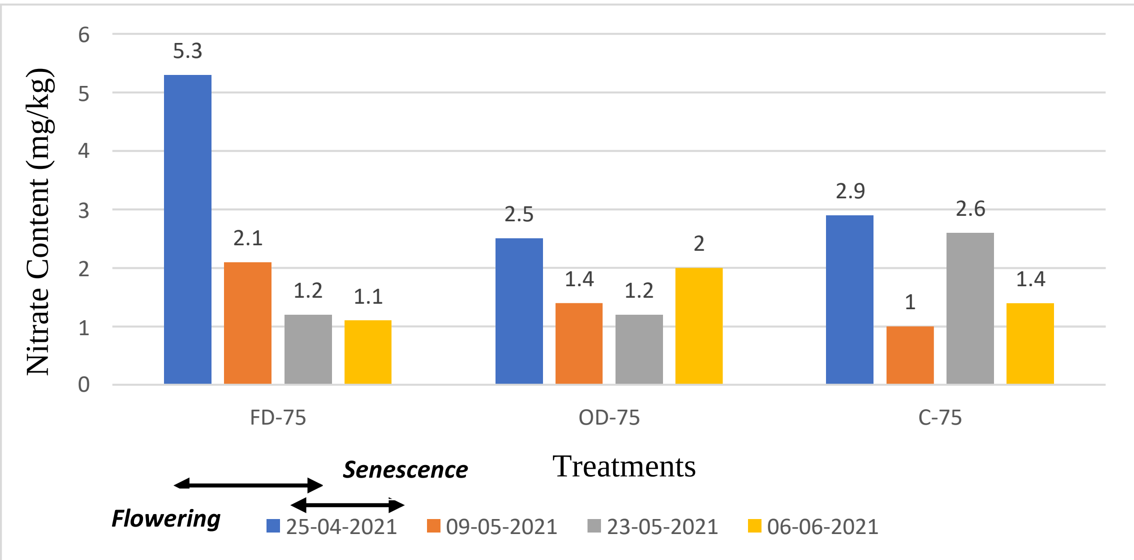

Figure 4.10: $NO_3^- - N$ content release curves from flowering to senescence growth stage with 75% AWC

4.3 Plant parameters

The results of various crop parameters, such as crop yield, plant height, stem diameter, leaf area index, plant biomass have been investigated and discussed in the following sections. In

Table 4.2, the statistical significance of numerous plant factors has been examined and tabulated.

Table 4.2: Significance of numerous plant parameters

Source	Crop yield	Plant height	Stem diameter	Leaf area index	Plant biomass	IWUE
Hydrogel treatment	*	N. S	*	N. S	*	*
AWC treatment	*	N. S	N. S	N. S	N. S	*
Hydrogel *AWC	*	N. S	N. S	N. S	N. S	*

(where * = significance at $p < 0.05$; N.S- not statistically significant)

4.3.1 Crop yield

The results, as depicted in Table 4.3, showed that the 95 % AWC (0.88 ± 0.19 kg plant^{-1}) produced the highest mean marketable yield in FDH treatments, while the ODH 95 % AWC treatment produced the lowest mean marketable yield (0.32 ± 0.02 kg plant^{-1}). The control treatment gave a higher yield than ODH, ranging from 0.4 ± 0.02 to 0.53 ± 0.02 kg plant $^{-1}$.

Table 4.3: Marketable yield (kg plant $^{-1}$) for various hydrogel-AWC treatment combinations

	Treatments		
AWC (%)	**FDH**	**ODH**	**Control**
95 AWC	0.88 ± 0.02^{a}	0.32 ± 0.02^{d}	0.40 ± 0.02^{c}
75 AWC	0.54 ± 0.02^{b}	0.39 ± 0.02^{c}	0.53 ± 0.02^{b}

$^{a\text{–}d}$*Means followed by the same letter within a column are not significantly different at* p = 0.05. *The values reported are an average of three replicates.*

The analysis of the study implies that FDH with 95% AWC are well-suited for better optimization of water and can be used in drought-like regions for tomato crops. The result is consistent with the findings of Lopes et al. (2017), which revealed that hydrogels could increase crop yields even in water-scarce conditions, thus giving higher yields compared to the control.

Calcagnile et al. (2019) suggested that the hydrogel material formulation is well suited for agriculture applications, with the highest yield, fruit count, and plant height compared to the control. Moreover, the reason for ODH to have the least number of fruits is due to the low swelling capacity of Oven-dried beads. This can be demonstrated by the study carried out by Oliva (2020), where ODH can re-swell 184.5% less than the FDH, hence can absorb less water and nutrients.

The statistical analysis showed that the crop yield recorded a coefficient of determination of 0.97, with average crop yield to be 0.51 ± 0.19 kg plant $^{-1}$. The interaction factor of the hydrogel treatment, control, and the AWC were found to be statistically significant ($p < 0.05$) with an F value of 86.70, implying that all of the combinations of hydrogel treatment (HT) (FDH, ODH), control treatment and AWC (75% AWC and 95% AWC) are statistically significant with $p < 0.05$. Moreover, all the treatment differences calculated were found to be statistically significant ($p < 0.05$).

4.3.2 Plant height

4.3.2.(a) Hydrogel treatments with 95 % available water content

The analysis for the plant height over the growing season showed that the highest values were seen for FDH and the control treatments, while ODH showed lower values in comparison with the FDH and control treatments. The plant height (cm) ranged from 17 ± 7.21 cm to 101 ± 7.21 cm for FDH and control treatments, while a range of 17 ± 7.21 -92 ± 7.21 cm was observed in the case of ODH (Figure 4.11). Moreover, during the time of vegetative growth stage, all the three treatments showed a similar plant height of 69.33 ± 7.21 cm, but a marginal change in the plant height was seen in the ODH treatments (92.17 cm), in comparison to other treatments (101.67 cm) after vegetative growth stage. The decline in the height of the crop had no significant impact on various hydrogel and AWC treatment-combinations, hence we can conclude that all the treatments behaved consistently for 95% AWC threshold.

4.3.2.(b) Hydrogel treatments with 75 % available water content

The plant height for 75% AWC during the 4-month tomato growth season is shown in Figure 4.12. It was observed that the plant height considerably increased over the growing season. All the treatments showed a similar range from 18 to 96.53 cm, and there is no noticeable difference observed amongst the treatment combinations for the 75% irrigation threshold.

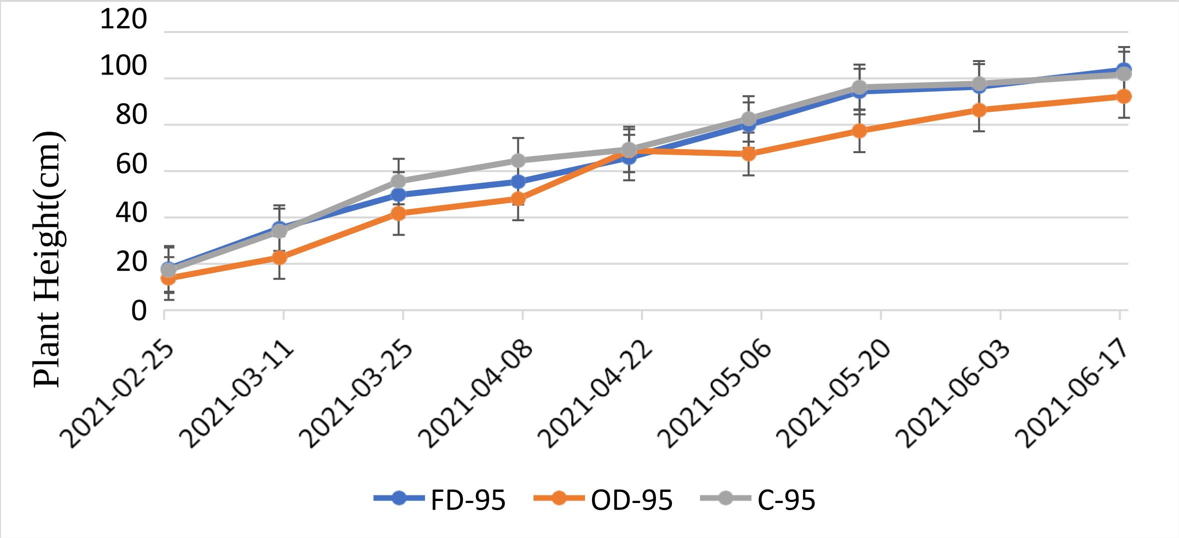

Figure 4.11: Plant height (cm) of crop comprising hydrogel treatments with 95% AWC

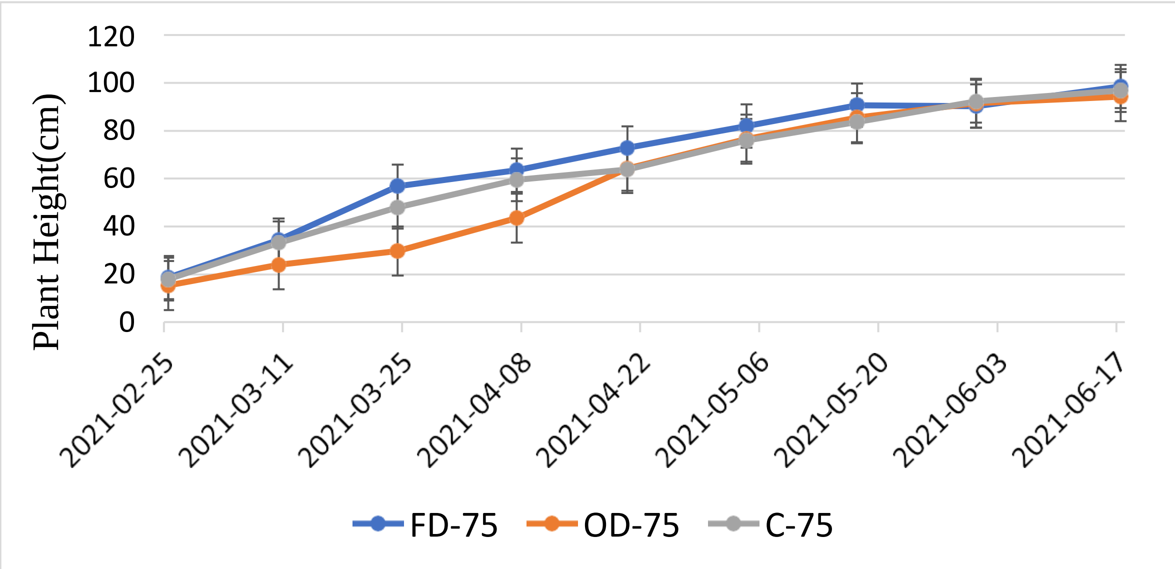

Figure 4.12: Plant Height (cm) of crop comprising hydrogel treatments with 75% AWC

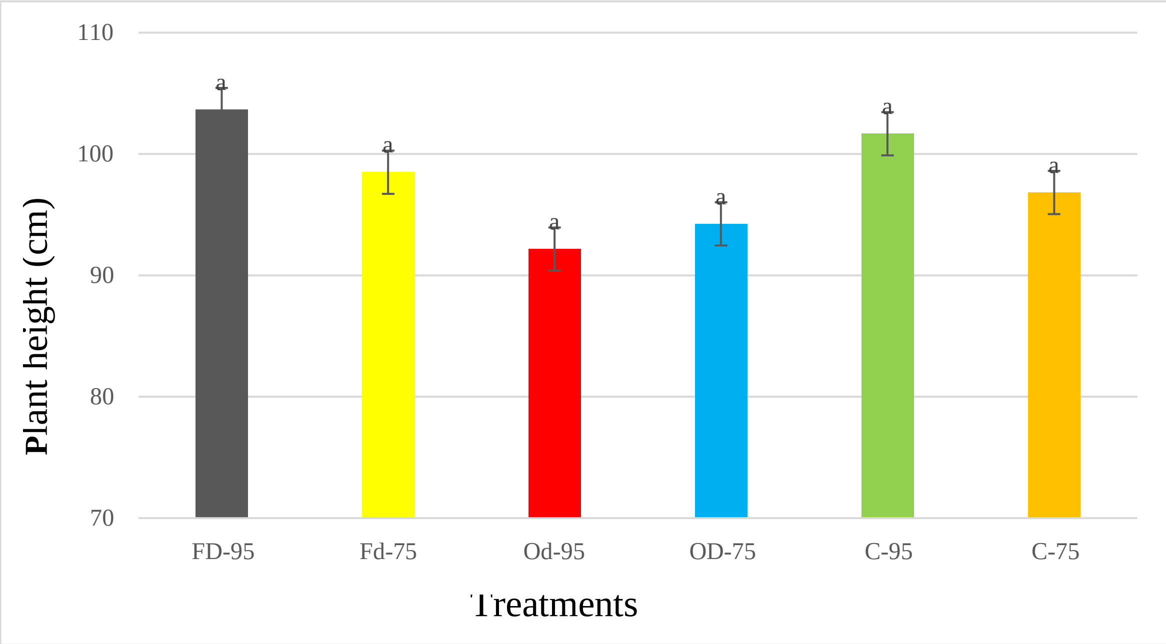

Figure 4.13: Comparison of plant height of various hydrogel and AWC treatment – combinations

Where, means followed by the same letter within a column are not significantly different at $p = 0.05$. *Reported values are averages of three replicates.*

The primary results of plant height showed that there was a negligible difference in the plant height for various hydrogel and AWC treatment – combinations, hence all of the combinations showed similar plant height. The findings of the study on ficus Benjamina starlight, carried out by Ghehsareh et al. (2010) are in the line with the research, showing similar results on the plant, height, stem number, leaf area ratio, which implies that these parameters are not impacted by the various treatment combinations. Similar results were found by Hernanzed et al. (2017), with the use of chitosan hydrogels, which showed that there was no significant difference in the plant height. Our findings differ from those of Montesano et al. (2015), which found that the application of cellulose hydrogels resulted in a 22-cm increase in plant height in the cucumber crop when compared to soil without hydrogels. However, the research carried out with freeze-dried, and oven-dried cellulose hydrogels is novel from agriculture point of view and are still under investigation.

Based on our statistical analysis, plant height shows a lower coefficient of determination of 0.13, between hydrogel, control, and AWC treatments, with a mean of approximately 97.85 ± 7.21 cm. This value of R^2 signifies that the plant height was not significantly impacted by the combination of hydrogel and AWC treatments, hence the combination of hydrogel and AWC, does not have a significant effect on the plant height (F value of 0.16 and P= 0.8532). Also, the differences in all of the treatment combinations were not statistically significant on the plant height ($p > 0.05$).

4.3.3 Stem diameter

The results of stem diameter over the growth period are illustrated in Figure 4.14. In general, the values of stem diameter increased from 10 ± 1.47 to 28 ± 1.47 mm, during the growth period. At the beginning of the vegetative growth, the approximate value stem diameter (23.01 ± 1.47mm) rose slightly till the end of the growth season (28.53 ± 1.47mm). For all of the treatment combinations, the majority of the stem was developed during the vegetative growth season, and there was no noticeable difference found after this stage (i.e., till 20th April 2021).

In comparison with the various treatments, FDH with both of the AWC showed better results as compared to the control and the ODH treatments. The reason FDH exhibited a slightly modest behavior, in comparison with the ODH and control treatments was due to the size and the morphology of the beads (Oliva, 2020). The higher swelling capacity of the beads embedded a positive impact on the overall performance of the plant growth. The range found for 95% AWC treatments was from 25 ± 1.47 to 28 ± 1.47 mm, while for 75% AWC it was in a range of 22 ± 1.47 -28 ± 1.47 mm.

Overall, there was a two-fold increase in the values of the stem diameter of all of the treatment combinations, from the day of transplanting to the day of harvest. The research is in line with the findings of Hernanzez et al. (2017), where the stem diameter of the plants increased with the application of chitosan-based natural hydrogels. Moreover, the findings of Kalhapure et al. (2016) suggest that with the increase in the dosage of the cellulose hydrogels, an increase in stem diameter, number of leaves, and branches were observed. Furthermore, increased porosity improves seed germination, rate of seedling emergence, root growth density, with reduced soil erosion due to reduction in soil compaction. Furthermore, Senna et al. (2017) reported that that average height and stem diameter growth of HEDTA cellulose hydrogels amended with NPK fertilizer enhanced the development of eucalyptus seedlings in Brazil during the dry season. These factors suggest that hydrogels can be useful for improving the crop productivity. However, Ghesareh et al. (2010) showed that the various treatments have no significant impact on stem diameter, hence there was no substantial difference between the hydrogel-treated samples and the control samples.

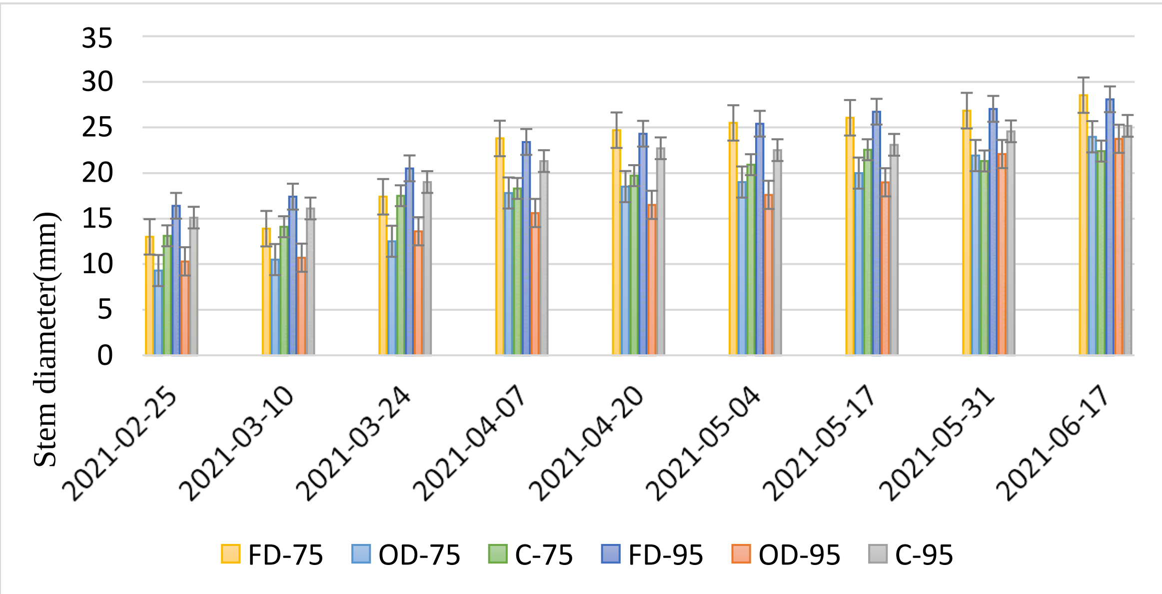

Figure 4.14: Stem Diameter (mm) of crop comprising hydrogel and AWC treatments

Statistically, stem diameter recorded a coefficient of determination of 0.54, with a mean of approximately 25.31 ± 1.47 mm of various hydrogel-AWC treatment combinations. The stem diameter is not impacted by the combination of hydrogel and AWC, hence the interaction factor of the hydrogel and AWC was not statistically significant (P=0.4979). As the combination of interaction (hydrogel*AWC) does not have a significant effect on the stem diameter, a further analysis was conducted. The investigation reported that hydrogel had a significant effect on stem diameter (F value of 6.18, and P =0.0143). Using the estimate statements in SAS 9.4, it was shown that the differences in various hydrogel combinations were found to be statistically significant ($p < 0.05$).

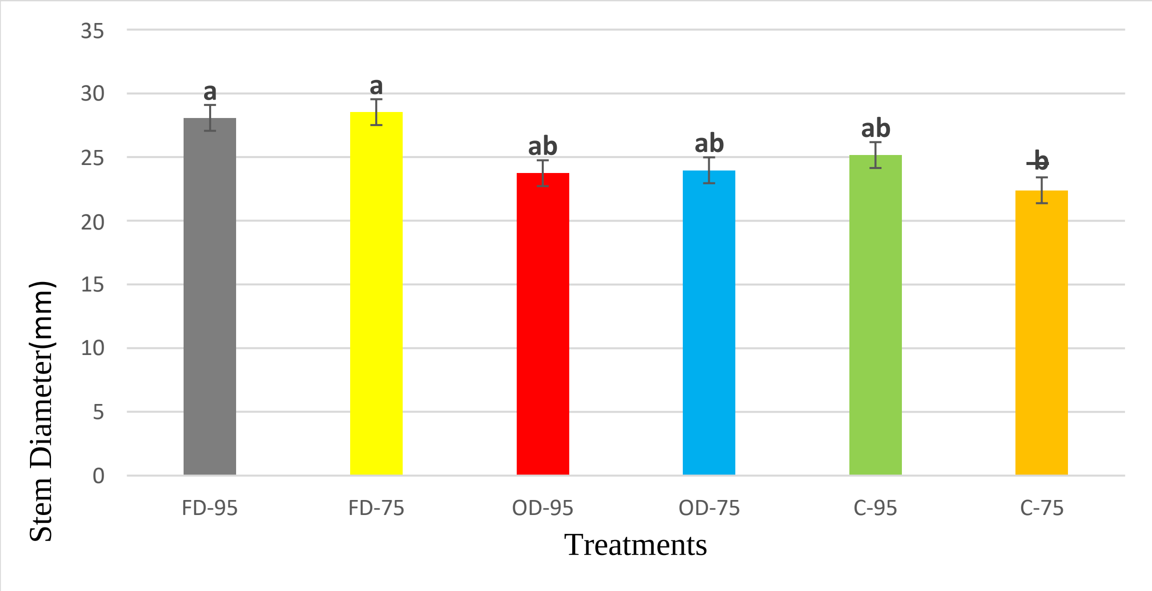

Figure 4.15: Comparison of stem diameter of various hydrogel and AWC treatment – combinations

Where, $^{a-b}$ means followed by the same letter within a column are not significantly different at $p = 0.05$. *Reported values are averages of three replicates.*

4.3.4 Total plant biomass

The results of total plant biomass of various treatments-combinations are shown in Figure 4.16. For 95% AWC, the FDH treatment showed the highest plant biomass of 2.00 kg plant^{-1}, while the ODH-75 treatments showed the lowest value of 1.03 kg plant^{-1}. The control-95% (1.59 kg plant^{-1}) AWC and 75% (1.69 kg plant^{-1}) AWC treatment was shown to have a higher value than ODH – 95% AWC (1.08 kg plant^{-1}) and 75% AWC (1.03 kg plant^{-1}) treatments. For the 75% AWC, the FDH and control treatments showed similar results of plant biomass of 1.65 kg plant^{-1}, while the ODH showed the lowest value of 1.03 kg plant^{-1}.

A study conducted by Orikiriza (2013) showed that the modification of soil with hydrogel can improve biomass production of tree seedlings, which is in line with this research. Furthermore, hydrogel improved the survival of the crop, before and after water stress conditions. As a result, the use of absorbent polymer could be encouraged to boost seedling production in both water-stressed and non-water-stressed conditions (Orikiriza et al., 2013). Our findings are in line with Montesano et al. (2015), who found that hydrogel-amended plants had higher overall fresh biomass (1753 g vs 913 g), leaf, stem, and fruit fresh biomass (468 vs 285 g, 427 vs 264, and 858 vs 364 correspondingly) than control plants.

Statistically, plant biomass recorded a coefficient of determination of 0.90, with a mean of approximately 1.70 ± 0.07 kg of various HC-AWC treatments. The plant biomass was not impacted by the combination of hydrogel and AWC, hence the interaction factor of hydrogel and AWC was not statistically significant (P=0.2643). As interaction (HC*AWC) was not statistically significant, another analysis was conducted to test the statistical effects of hydrogel and AWC on plant biomass. The findings of the analysis demonstrated that the AWC do not have a significant effect on plant biomass i.e., $p > 0.05$, but hydrogel treatments have a significant impact on the plant biomass of the crop ($p < 0.05$).

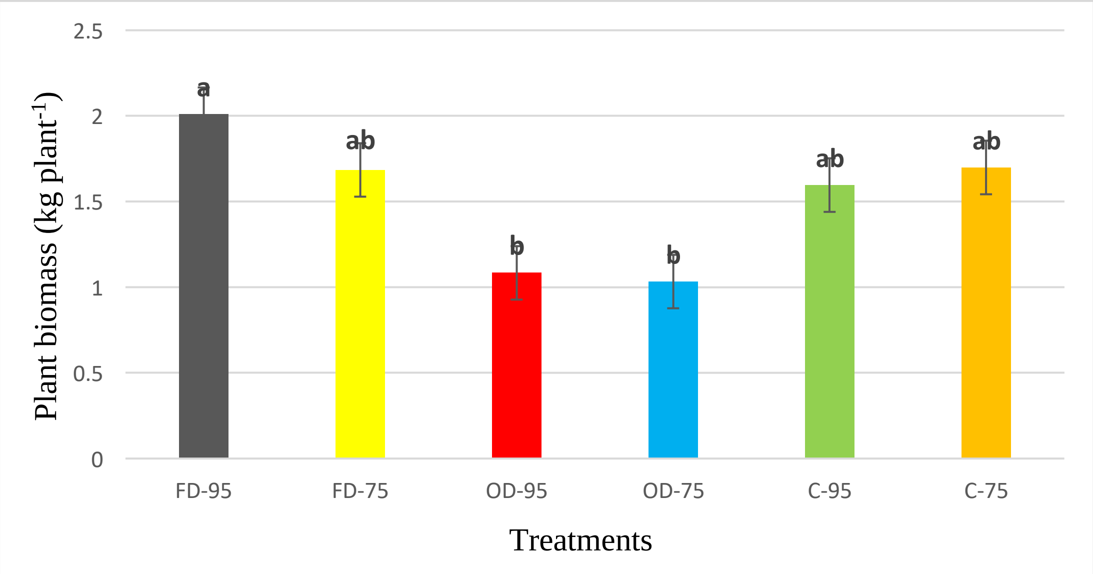

Figure 4.16: Plant biomass of various hydrogel and AWC treatment – combinations.

Where, [a-c]means followed by the same letter within a column are not significantly different at $p = 0.05$. *Reported values are averages of three replicates.*

4.3.5 Leaf area index

The LAI of FDH, ODH, and control treatments with respect to AWC are shown in Figure 4.17. The highest LAI was found in FD-95 (107.014 cm^2), followed by OD-95 of 103.991 cm^2. The LAI of C-95 (90.558) was found to be comparatively less than the other 95% AWC treatments. Moreover, the results for 75% irrigation thresholds, showed that the FD (95.120 cm^2) and OD (92.309 cm^2) treatments showed a higher LAI in comparison with the control treatment of 95 and 75% AWC. Hence, with the application of less water, higher values of LAI can be achieved in the case of control treatments.

With the application of cellulose hydrogel in the soil, Sasmal and Patra (2020) discovered that increased soil water holding capacity of the hydrogels resulted in improved corn seed germination and seedling growth in the amended soil. The findings by Sasmal and Patra, (2020) are in line with our study, where the hydrogel amended soil, had larger leaf area, resulting in more photosynthetic capacity of the older leaves with a greater number of chloroplasts per cell (Bauer and Thoni, 1988). Increased photosynthetic capacity of leaves can result in more fruits, as the efficiency with which a crop absorbs light and convert it into biomass throughout the growing season is a primary predictor of final yield (Long et al., 2006).

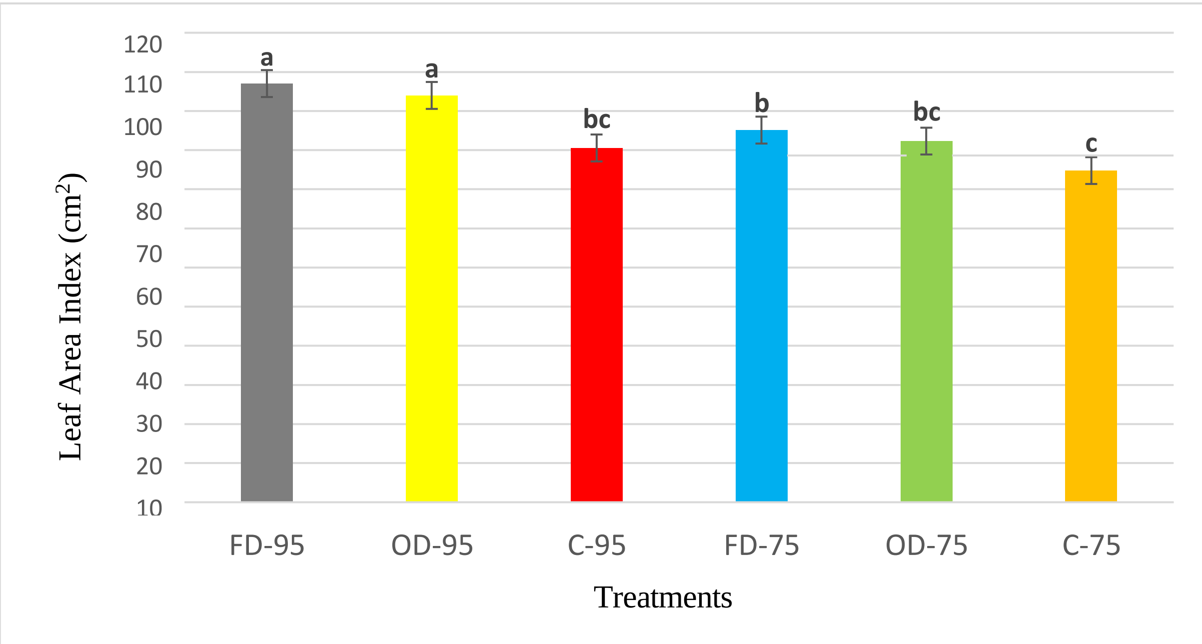

Figure 4.17: Leaf Area Index (LAI) of various hydrogel and AWC treatment – combinations

Where, $^{a-c}$means followed by the same letter within a column are not significantly different at $p = 0.05$. *Reported values are averages of three replicates.*

Statistically, LAI yielded a coefficient of determination of 0.894, with a mean of approximately 95.91 ± 3.69 cm^2 of various hydrogel and AWC treatments. The LAI is not impacted by the combination of hydrogel and AWC, hence the interaction of hydrogel and AWC factors was not statistically significant ($P = 0.4325$) i.e., $p > 0.05$.

4.3.6 Irrigation water use efficiency

The highest IWUE value of 3.911 kg $m^{-1} plant^{-1}$) was recorded with FDH-95, as shown in Table 4.3, while the lowest IWUE was reported by ODH-95 to be around, 1.467 kg $m^{-1} plant^{-1}$. The prime reason for oven-dried to show poor performance in comparison to freeze-dried hydrogels is due to the morphology of the oven-dried hydrogel beads retaining 185.4% less water as compared to the freeze-dried hydrogels.

Application of water at 75% AWC yielded a loss of 22.4% compared to the highest IWUE, demonstrating the necessity to apply more water to lower irrigation thresholds. The findings of this study are in line with various other authors (Ihoma and Madramootoo, 2019; Jaria and Madramootoo,2013; Petropoulos et al; 2019), who revealed that water stress can negatively affect the physiological parameters and the photosynthetic activity of the tomato crop. Tyagi et al. (2015) reported that the amendment of soil with hydrogels can increase the water use efficiency of wheat crops by an increase in water holding capacity and available water content. Various authors such as Koupai et al. (2008), Andry et al. (2009), and Dorraji et al. (2010) also stated that there is a drastic rise in WUE with the amendment of water-absorbing polymers in crops.

The statistical analysis showed that the IWUE recorded a coefficient of determination of 0.86, with an average IWUE of 2.48 ± 0.16 kg $m^{-1} plant^{-1}$. The interaction factor of the hydrogel treatment and the AWC was found to be statistically significant ($p < 0.05$) with an F value of 11.00, implying that all of the combinations of hydrogel treatment (HT) (FDH, ODH), control treatment (CT) and AWC (75% AWC and 95% AWC) are statistically significant with $p < 0.05$. Moreover, all the treatment differences calculated were found to be statistically significant ($p < 0.05$).

Table 4.4: Irrigation water use efficiency for various hydrogel treatment combinations

Treatment combination	**Marketable yield (kg plant $^{-1}$)**	**Depth of irrigation (m)**	**Irrigation Water use efficiency (IWUE) (kg/m-plant)**
FDH-95	0.88	0.225	3.911 ± 0.05^{a}
ODH-95	0.32	0.218	1.467 ± 0.05^{d}
CO-95	0.40	0.265	1.509 ± 0.05^{d}
FDH-75	0.54	0.178	3.033 ± 0.05^{b}
ODH-75	0.39	0.173	2.254 ± 0.05^{c}
CO-75	0.53	0.195	2.717 ± 0.05^{bc}

$^{a–d}$Means followed by the same letter within a column are not significantly different at $p = 0.05$.

Reported values are averages of three replicates.

Chapter 5 : Conclusions and Recommendations

5.1 Conclusions

Irrigation and fertilizer application are the two most important agronomic practices with potential of reducing input costs and improving crop yield. Current water scarcity and environmental concerns from over fertigation have intensified the need for irrigation and fertilizer application optimization. Therefore, new techniques for irrigation and fertilizer deployment to crop production are urgently required to conserve scarce resources and protect natural environment. Through the present study an innovative research project was undertaken to investigate the use of cellulose biodegradable hydrogels for their water and fertilizer retention properties and effect on crop yield. These hydrogels were derived from paper waste, which can retain and gradually release a significant amount of water and nitrogen. The objective of this study was to observe the crop response to water and fertilizers applied to soils modified with cellulose-based hydrogels. The experiment was conducted in a controlled greenhouse environment. The test crop was tomato. There were two hydrogel treatments (freeze-dried and oven-dried encapsulated with 20-20-20 N-P-K fertilizer) and two irrigation treatments i.e., 75% AWC and 95% AWC. The measured and calculated parameters include moisture content, nitrate content, crop yield, plant height, stem diameter, leaf area index, plant biomass, and irrigation water use efficiency.

The following conclusions were drawn as aligned with the objectives of the present study.

(i) FD hydrogels were found to be the most effective slow fertilizer releasing polymers as they retained, absorbed, and released nutrients, with a 3-fold increase in soil $NO_3^-{-}N$ content from 21.30 to 66.53 mg/kg, over a month.

(ii) Amongst the various hydrogel and AWC treatment combinations, FDH-95% AWC treatments showed the highest amount of soil $NO_3^- - N$ content at the start of the growing season. This soil nitrate was gradually released and absorbed by the plants.

(iii) ODH and control treatments (without hydrogels) were found to be uneven in the soil nitrate concentration, due to net absorption and assimilation rate in plants.

(iv) Freeze dried hydrogels with 95% AWC exhibited a statistically significant difference, compared to the other treatments, with the highest total marketable yield of 0.88 ± 0.02 g plant^{-1}.

(v) Hydrogel treated samples had less irrigation water requirement and higher yield, in comparison with the control. Hence the adaptability of hydrogels in arid and semi-arid environments would be extremely beneficial.

(vi) None of the hydrogel and AWC treatment-combinations showed a statistically significant effect on plant height and stem diameter of the tomato crop. Whereas FD-95% AWC showed a statistically significantly higher leaf area index and plant biomass compared to other hydrogel and AWC combinations.

(vii) It was found that FDH-95% had the highest IWUE of 3.911 ± 0.05 kg m^{-1}plant^{-1}, compared to other treatments.

5.2 Recommendations

Based on the findings of this study, the following recommendations are drawn:

1) More long-term field and greenhouse studies could be done with different crops, to better understand crop response to cellulose-based hydrogels.

2) Measurement of tomato fruit quality parameters (lycopene, flavonoids, phenol content, antioxidant activity) under different hydrogels, irrigation treatments and soil types should be assessed.

3) Investigation and comparison of other biodegradable hydrogels (chitosan and alginate-based) treated by freeze drying, oven drying can be undertaken.

4) Using long term studies, various hydrogel parameters should be evaluated such as; equilibrium swelling ratio over time, the biodegradability of hydrogels, changes in the morphology of the beads using scanning electron microscopy.

5) Expanding the scope to manufacture the hydrogels using environment-friendly chemicals other than NaOH/urea. The applicability of the manufactured hydrogels can be tested to various crops in greenhouse and field studies.

6) Investigation of other drying mechanisms such as spray drying and supercritical drying mechanisms of cellulose-based hydrogels and assessing their effects on various soil types and crops.

Chapter 6 : References

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