Alternative to Environmental Pollution Plastics: Production of Hydrogel Film Using Clove Plant Extract and Controlling it with a Robotic System

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ABSTRACT

Although petroleum derived synthetic plastics have important advantages, they cause ecological pollution because they are produced more than 250 million tons per. Biodegradable packaging has emerged as an alternative to petroleum-derived plastics. In this project, a robotic system was combined with hydrogels produced to extend the shelf life of food. In the study, after hydrogels were produced with 5 extracts as 0%(control), 12.5%, 25%, 50%, 100%, the water holding capacity, water solubility, water vapor permeability of the hydrogels were examined. aromaticum extract increased.

Key words: *hydrogels, petroleum, ecological pollution, robotic system*

1 Introduction

The macromolecules formed by many small molecules bound together are called polymers. Polymers are formed by the chemical bond that monomers form as a result of the reaction under suitable conditions. Polymers are studied as natural and artificial. In the natural polymers section, proteins, cellulose, chitin, starch, RNA and DNA; In the artificial polymers section, there are synthetic rubber, PVC, silicone, nylon and plastics (Saçak, 1998). Some artificial polymers are widely used in packaging (Sarıgül, 2018).

Packaging, in the most general sense, can be defined as wraps and containers that protect the products included in the distribution process from environmental factors in the distribution process, keep the product as a whole, and facilitate transportation, distribution, storage and promotion. Having an important place in the food industry, packages ensure that foodstuffs are delivered to consumers in a healthy way. However, the main reason that makes the packaging important for the food sector is its importance for public health. If the foods prepared in healthy conditions are not packaged correctly, the foods become spoiled and threaten the health. Therefore, the right packaging and packaging selection is important (Öztürk, 2020). Thus, it is possible to keep food for a long time without spoiling.

One of the most used products as packaging is plastics. Plastics have become lighter, more sophisticated and versatile with innovative applications in recent years, and have replaced traditional packaging products such as paper and glass. In the past, materials such as cellophane transparent cellulose film and cellulose acetate were used in addition to traditional packaging such as wood, glass and paper, while plastic packaging began to be used extensively with the production and introduction of polyethylene in the 1950s. With the production of PVC, polystyrene, polyester, polypropylene and polyethylene copolymers, plastics have become increasingly common in the packaging industry (PAGEV, 2019). The important advantages of plastics that are used so heavily are that they are lightweight, they are durable, they are safe when used as containers, they are easy to shape, they are flexible, they have good insulation, they can be used for moist foods and they are suitable for use in microwave ovens (Güler and Çobanoğlu, 1997). Despite its advantages, approximately one-third of the petroleum-derived synthetic plastics, which are produced more than 250 million tons per year and are widely used, are used as packaging, then left to nature in the form of waste, and the processes of fragmentation and mixing into the soil negatively affect the living things. These wastes, especially plastic bags, are blown away from the area where they are left by the wind and drifted to different regions or they are transported by rivers and cause visual pollution and pollution of living spaces. For this reason, petroleum-based synthetic plastics have become an important environmental problem today. Since oil is not a renewable resource and synthetic plastics increase dependence on oil, it is a serious problem (Bölükbaşı, 2012).

With the understanding of the harmful effects of petroleum-derived synthetic plastics to the nature, biodegradable polymers of biological origin have started to be used more in plastic production in recent years (Şahin, Demir, İlsay, & Doğdubay, 2017). Biodegradable plastics; These are plastics produced from renewable sources such as plant starches and cellulose (Mehmet KILINÇ, Oktay TOMAR, Abdullah ÇAĞLAR, 2017). Biodegradable plastics obtained from different sources have some advantages over synthetic plastics. These; It can be listed as reducing dependence on oil, providing energy efficiency, causing less greenhouse gas emissions during production, and being more environmentally friendly since they do not contain toxic substances. Despite these advantages, it also has some disadvantages. These are; The cost is higher than synthetic plastics, recycling problems and the use of polymers such as starch-protein in the production process are seen as a disadvantage as these polymers meet the nutritional needs of the world population (Köksal, Aydın Er, Adalı, & Sağlam, 2019). When these disadvantages are overcome, it can be predicted that biodegradable plastics will take an important place in the plastics industry. However, biodegradable plastics used in different sectors account for 1.5% of total plastic production (PAGEV, 2020). However, the global market for biodegradable plastics is expected to grow further in the coming years. According to the latest market data compiled in collaboration with Nova-Institute and European Bioplastics, global production capacities of biodegradable plastics are estimated to increase from approximately 2.11

million tons in 2019 to approximately 2.42 million tons by 2024.

Some of the hydrogels are in the biodegradable polymers class (Çelik & Tümer, 2016). Hydrogels are used in the production of artificial retina and muscle in the health sector (Güngör & Erkan, 2004). In the literature, it has been observed that hydrogels are generally used in the health sector, and there are also studies on their use as packaging (Şahiner, Sağbaş, Turan, Erdugan, & Şahiner, 2018).

The food industry is a sector with a lot of plastic packaging waste, which creates a huge ecological problem. The food sector is also intertwined with the electronics sector, because people are now more sensitive about the packaging conditions of food, the place of production and date, and at the same time, many different advertising strategies are applied in the food sector (Aydın, 2012). In this project, we decided to use arduino to measure some parameters we have determined in order to unite the food and electronics sectors.

Many different projects can be carried out due to the fact that the open source equipped and software Ardunio controllers, which have been widely used in recent years, can detect some chemical parameters with additional sensors. The Ardunio platform enables work to be carried out more cheaply and quickly (Dipova, 2017). Ardunio has types that appeal to many different functions. Some of these are UNO for small-scale projects, MEGA for larger projects, and LILYPAD for use on clothing. (10) NodeMCU creates a platform that enables the Internet of Things (iot) to be remotely detected, connected and controlled by devices via a network server. It is known that many physical objects cannot be connected to the network. Thanks to the Internet of Things, inter-machine communication technologies and ecosystem, it is aimed to control and monitor these objects on the network.

In this project, we aim to observe the effect of hydrogels on the shelf life of foods using Arduino.

1- 1 Aim

The aim of this study is to synthesize hydrogel films using clove (Syzygium aromaticum) extract, to examine the properties of these films, to determine their usability as nutrition storage material, and to create a system that provides information about the state and shelf life of nutrition by obtaining some information on the environment through a system created with Arduino and sensors. The investigated features are; microscopic images of films, their thickness, water solubility, water vapor permeability, water holding capacity and antibacterial properties, and their effects on extending nutrition shelf life. If the synthesized films have the properties required for preserving nutrition, these materials will be important in terms of protecting the nature as they will be an alternative to petroleum-based plastics that dissolve in nature in a very long time. If these materials have antibacterial properties and effects to extend the shelf life of nutrition, they will be suitable materials to preserve nutritions in a healthy way. In addition, taking certain features of the environment in an environment created with the Arduino system will contribute to gaining a healthy idea about the characteristics of the indoor environment without opening the nutrition.

2 Method

2- 1 Devices Used in Experimental Procedures

The devices used in the study; analytical balance, magnetic stirrer, ultrasonic water bath, Soxhlet extraction device, oven, caliper and McFarland densitometer.

2- 2 Production of Hydrogel Films

The devices used in the study; analytical balance, magnetic stirrer, ultrasonic water bath, Soxhlet extraction device, oven, caliper and McFarland densitometer.

50 g clove plants were extracted in 500 ml purified water using Soxhlet extractor for 2 hours. The extract obtained at the end of 2 hours was made ready to be used in the production of hydrogel films. Hydrogel films were produced by the method of dissolving-drying, taking into account the proportions used by Topdağ (2015) in his study. In the production of each film, sodium alginate measured by analytical balance at 0.2 g was taken into a beaker. By adding 10 ml of distilled water on it, it was heated in a magnetic stirrer at 600C at 600 rpm for 1 hour and then dissolved completely. The solution obtained was cooled down to 25 ° C and 39 µl of glycerol was added on it and kept in an ultrasonic water bath for 15 minutes. The prepared mixtures were kept in ultrasonic water bath for 15 minutes, then poured into petri dishes and dried for 24 hours. 0.2 M $CaCl_2$ solution was poured on the dried films and kept for 5 minutes. At the end of this period, the films were washed with distilled water for 5 minutes and dried at room conditions to obtain hydrogel films. The control group (0% extract) hydrogel film was synthesized in this way. In other examples, clove (Syzygium aromaticum) extract concentrations were added instead of water in the part where the alginate was dissolved in water, at concentrations of 0%, 12.5%, 25%, 50% and 100%.

2-3 Examination of Hydrogel Film Properties

2- 3- 1 Examining the Thickness of Hydrogel Films

Calipers were used to measure the thickness of the hydrogel films. Measurements were made by taking three samples for each film, and the results were presented on average values.

2-3-2 Examination of Hydrogel Films' Water Vapor Permeability

Water vapor permeability of hydrogel films was determined using ASTM E96 / E96M standards. After adding 5 grams (W1) of pure water to each Falcon tubes, the films were glued to the mouth of the tubes and the edges were wrapped with Teflon tape. The samples were stored in the oven at 25°C for 24 hours and the remaining water (W2) was weighed. (A= surface area of falcon tubes). The water vapor permeability of hydrogel films was calculated (Eq. 1).

$$Water\ vapor\ permeability = \frac{W1 - W2}{A} \quad (1)$$

The experiments were repeated with three films for each concentration.

2-3-3 Examination of Hydrogel Films' Water Solubility

In order to examine the water solubility of hydrogel films, the (W0) films weighed before the experiment were left in a beaker containing 20 ml of distilled water and kept in a magnetic stirrer at 120 rpm at 25°C for 24 hours. The films were then removed from the water, dried with filter paper and reweighed (W1). Then, the water solubility of the films was calculated(Eq. 2).

$$Water\ solubility = \frac{W0 - W1}{W1} x100 \quad (2)$$

The experiments were repeated with three films for each

2-3-4 Examination of Hydrogel Films' Water Storing Capacity

In order to determine the water retention properties of hydrogel films, first the dry films were weighed (W0), then they were thrown into pure water and kept in the oven at 25ºC for 24 hours. The films were then removed from the container and dried on filter paper for 5 minutes and the films were weighed (W1). The water holding capacity of the films was calculated (Eq. 3).

$$\text{Water storing capacity} = \frac{W1-W0}{W0} x100 \qquad (3)$$

2-3-5 Investigation of Antibacterial Properties of Clove (Syzygium Aromaticum) Extract used in Hydrogel Films

To examine the antibacterial properties of the clove (Syzygium aromaticum) extract used in hydrogel films, gram-positive bacteria Staphylococcus aureus and a gram-negative bacterium Escherichia coli were used and the zone diameters of the extract were determined by agar diffusion method. After the bacteria were obtained from the university in our city, they were incubated at 37ºC for 24 hours in the medium before the study and the study was carried out with fresh bacteria. Bacteria were diluted to 0.5 McFarland using a McFarland Densitometer. After the prepared bacterial suspension was added to the media, wells with a diameter of 6 mm were opened in the middle of the media and 50 µL of extracts of different concentrations were added. After the bacteria in the media were incubated in the oven at 37ºC for 24 hours, the zone diameters formed in the medium were measured.

2-4 Monitoring and Investigation of the Decay Amount of the Food Wrapped in Hydrogel Film with Arduino System

MQ-2 (Gas Sensor) and DHT11 (temperature and humidity sensor) sensors were used to examine the spoilage amount of food wrapped in hydrogel film. The values read from the sensors used were transferred to the Blynk application over the internet via the Blynk library and the ESP-8266 Wifi module. The transferred data was checked and analyzed with the time-value graph on the application. As long as the ESP-8266 Wifi module is connected to the internet, a system has been created that can examine the data transferred from the Blynk application from any point in the world. The condition of the food wrapped in Hydrogel film was monitored with the system created.

2-5 Control of Films with Robotic System

2-5-1 Examination of Gas Amount in Closed Environment with MQ-2 Gas Sensor

In order to determine the shelf life of the food placed in the closed environment, the gas amount in the system was measured with the MQ-2 Gas sensor. When the obtained values are examined, the increase in gas in the system can be used to understand that there is an increase in bacteria. Because as the number of bacteria in the environment increases, the amount of gas produced as a result of the vital activities of the bacteria is expected to increase.

2-5-2 Inspection of Indoor Temperature with DHT-11 Temperature-Humidity Sensor

In order to estimate the shelf life of the food placed in the closed environment, the temperature of the environment was measured with the DHT-11 Temperature-Humidity sensor. When the obtained values are examined, the temperature increase can be used to understand that there is an increase in bacteria. It is expected that the vital activities performed by the bacteria will cause an increase in the ambient temperature. Bacteria and the increase in temperature in the environment are also one of the important causes of food spoilage.

Monitoring and Examining the Data Collected in the System over the Internet. The data obtained from the temperature-humidity and gas sensor were monitored over the internet using the Blynk application and library via the Arduino NodeMCU Wifi card. The collected data were analyzed in graphics and with SPSS (Fig.1).

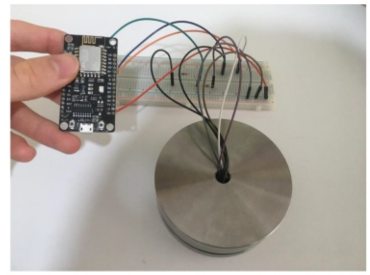

Fig. 1 : The Robotic System

3 Results and Findings

3-1 Results for Examining the Thickness of Hydrogel Films

Calipers were used to measure the thickness of the hydrogel films and average film thicknesses were calculated by taking three samples from each film. The measured film thicknesses are presented in Table (1).

Table (1) : Results regarding the thickness of hydrogel films

Extract Concentration Used in Film	Film Thickness (µm)
%100	208
%50	202
%25	212
%12,5	215
%0 (Control)	209

When Table 1 is examined, it is seen that the thicknesses of the films (for 100% -50% -25% -12.5% -0% (control) extract concentrations) are 208 µm, 202 µm, 212 µm, 215 µm and 209 µm, respectively.

3-2 Results for Investigation of Water Vapor Permeability of Films:

The measured values obtained to examine the water vapor permeability of hydrogel films and the calculated water vapor permeability of the films are presented in Table (2).

Table (2): Results regarding the water vapor permeability of hydrogel films

Extract Concentration Used in Film	W1 (g)	W2 (g)	A (m^2)	Water vapor permeability
%100	5.0554	4.6169	0.000314	1396.4968
%50	5.0193	4.5891	0.000314	1370.0637
%25	5.0315	4.6357	0.000314	1260.5095
%12,5	5.0358	4.6992	0.000314	1071.9745
%0 (Control)	5.0112	4.7713	0.000314	764.0127

When Table (2) is examined, the sample with the highest water vapor permeability is the sample with a concentration of 100%. Him respectively; 50% 25%; It is followed by films containing 12.5% and 0% (Control) Syzygium aromaticum (Clove) extract concentrations. The findings show that the addition of Syzygium aromaticum (Clove) extract to the films increases the water vapor permeability.

3-3 Results for Investigation of Water Solubility of Hydrogel Films

The measured values obtained to examine the water solubility of hydrogel films and the calculated water solubility of the films are presented in Table (3).

Table (3): Results regarding the solubility in water of hydrogel films

Extract Concentration Used in Film	W0 (g)	W1 (g)	Solubility in Water (%)
%100	0.0697	0.0628	9.90
%50	0.1366	0.1231	9.88
%25	0.1256	0.1133	9.79
%12,5	0.0946	0.0856	9.51
%0 (Control)	0.1458	0.1442	1.10

When Table (3) is examined, the findings obtained in the water solubility analysis of hydrogel films prepared with Syzygium aromaticum extract at different concentrations show that the water solubility of the films increases as the extract concentration increases. While the least soluble in water is the hydrogel film with a concentration of 0%, it is the most soluble film with a concentration of 100%.

3- 4 Results for the Investigation of the Water Holding Capacity of Hydrogel Films

The measured values obtained to examine the water holding capacity of hydrogel films and the calculated water holding capacity of the films are presented in Table (4).

Table (4): Results regarding the water holding capacity of hydrogel films

Extract Concentration Used in Film	W0 (g)	W1 (g)	Water Holding Capacity (%)
%100	0.0229	0.0697	204.37
%50	0.0133	0.0374	181.20
%25	0.0171	0.0439	156.72
%12,5	0.0157	0.0335	113.38
%0 (Control)	0.0190	0.0390	105.26

When Table (4) is examined, it shows that the hydrogel films prepared with Syzygium aromaticum (Clove) extract in different concentrations have the best water holding capacity, the one with 100% concentration. It was followed by 50%, 25%, 12.5% and 0%, respectively. As the concentration increases, the water holding capacity increases.

3-5 Results of the Antibacterial Properties of Syzygium aromaticum (Clove) Extract Used in Hydrogel Films

In order to examine the antibacterial properties of the extract obtained from Syzygium aromaticum (Clove) plant, bacteria were cultivated, 60 µl of extract was placed in 6 mm diameter wells and the zone diameters formed in the media after 24 hours of incubation were measured. The findings obtained are presented in Table (5).

Table (5). Results regarding the zone diameter for E. coli

Extract Concentration Used in Film	Zone Diameter for E. coli(mm)
%100	28
%50	24
%25	22
%12,5	19
%0 (Control)	6

When Table (5) is examined, the zone diameters of different concentrations of Syzygium aromaticum (Clove) extract (0%, 12.5%, 25%, 50% and 100%) in S. aureus and the zone diameters formed in the media in which E. coli are cultivated are 6 mm, 19 mm, 22 mm, 24 mm and 28 mm, respectively. It is seen that the zone diameter formed in the medium increases as the extract concentration increases.

3-6 Results of Investigation of Values from MQ-2 Gas Sensor and DHT-11 Temperature Sensors

When the water holding capacity, water vapor permeability, water solubility and antibacterial properties are examined, it has been seen that the hydrogel film containing 100% extract is a more suitable material for food storage than the others. Therefore, by using gas and temperature sensors, a closed environment in which 100% extract-containing hydrogel film is used was created, and measurements related to the food placed in this environment were taken, and as a control group, measurements of the food in an environment that did not use hydrogel film were compared. For the experimental group, the foods wrapped in hydrogel were kept in a closed container for 1 week. For the control group, the foods that were not wrapped in hydrogel were kept in a closed container for 1 week. The values taken from the temperature and gas sensors have been converted into tables (The values given by the sensors are numerical values that indicate the gas density or temperature change in the environment and are not expressed according to any unit. Therefore, the unit is avoided in the tables created. This is due to the presence of a single analog input on the NodeMCU. One of the circuit methods used when solving the problem of getting values from 2 sensors is to get values in Celcius format from the temperature sensor with the DHT-11 library) (Table 6).

Table (6): Values from MQ-2 and DHT-11 sensors for the experimental group

MQ-2	DHT-11	Day	Hour
163	191	Day 1 Morning	9.00
168	190	Day 1 Noon	12.00
175	192	Day 1 Evening	21.00
182	190	Day 2 Morning	9.00
186	191	Day 2 Noon	12.00
195	193	Day 2 Evening	21.00
203	193	Day 3 Morning	9.00
215	194	Day 3 Noon	12.00
232	193	Day 3 Evening	21.00
245	192	Day 4 Morning	9.00
261	196	Day 4 Noon	12.00
294	203	Day 4 Evening	21.00
314	211	Day 5 Morning	09.00
327	215	Day 5 Noon	12.00
331	219	Day 5 Evening	21.00
333	221	Day 6 Morning	09.00
337	222	Day 6 Noon	12.00
341	223	Day 6 Evening	21.00
339	221	Day 7 Morning	09.00
340	224	Day 7 Noon	12.00
342	223	Day 7 Evening	21.00

Table (7): Values from MQ-2 and DHT-11 sensors for the control group

MQ-2	DHT-11	Day	Hour
148	194	Day 1 Morning	9.00
153	195	Day 1 Noon	12.00
184	195	Day 1 Evening	21.00
202	198	Day 2 Morning	9.00
217	203	Day 2 Noon	12.00
235	209	Day 2 Evening	21.00
243	234	Day 3 Morning	9.00
231	246	Day 3 Noon	12.00
242	259	Day 3 Evening	21.00
321	267	Day 4 Morning	9.00
348	257	Day 4 Noon	12.00
336	256	Day 4 Evening	21.00
337	248	Day 5 Morning	09.00
335	251	Day 5 Noon	12.00
338	242	Day 5 Evening	21.00
335	231	Day 6 Morning	09.00
337	241	Day 6 Noon	12.00
336	224	Day 6 Evening	21.00
330	211	Day 7 Morning	09.00
324	225	Day 7 Noon	12.00
341	220	Day 7 Evening	21.00

When the tables were examined, when the results obtained from the experimental group were compared with the control group, it was observed that the temperature and gas increase in the experimental group was slower in the environment where the food was present. The data obtained from the experimental and control groups were compared with the t-test using the SPSS 22 program. The values obtained from the analysis of the obtained data with the t-test are presented in Table (8).

Table (8): T-test results of data obtained from MQ-2 and DHT-11 sensors in experimental and control groups

Sensor	Group	N	Ort.	SS	Sd	t	p
MQ-2	Example	21	263.000	69.9157	40	-.775	.443
	Control	21	279.667	69.5358			
DHT-11	Example	21	204.619	14.0658	40	-4.015	.000
	Control	21	228.857	23.8250			

When Table (8) is examined, it is seen that the mean temperature and gas density for both temperature and gas sensors are higher in the control group than in the experimental group. This situation shows that the temperature of the environment and the gas density in the environment increase when the food is placed in the environment without being wrapped in hydrogel film, in terms of the values obtained from both sensors. The increase in temperature and gas density in the environment is an important reason for food to spoil more easily. When the t-test was performed to examine whether the increase in temperature and gas density in the experimental and control groups was statistically significant, it was seen that the difference between the experimental and control groups was not significant for the gas sensor, but was significant for the temperature sensor. Although the difference between the groups in the gas sensor is not statistically significant, it should not be ignored that the mean of the control group is higher. When the values taken from both sensors and the statistical analysis are evaluated together, it can be said that the environment in which the hydrogel film produced can be a longer-term storage environment for foods.

When the studies in the literature are examined, food industry wastes (Yu, Chua, Huang, Lo and Chen, 1998), tequila pulp (Alva Munoz and Riley, 2008), corn cob (Bölükbaşı, 2012), algae (Özdemir and Erkmen, 2013; Civelek Yörüklü). , 2020), tea factory waste (Ersoy and Sutay Kocabaş, 2014), pomace (Demir and Sutay Kocabaş, 2014), fatty acid waste (Reddy, Amulya, Rohit, Sarma and Mohan, 2014), beverage waste (Elçiçek and Tanyıldızı, 2015), wastewater (Amulya, Reddy, Rohit, and Mohan, 2016; Yadav, Pandey, Kumar, and Tyagi, 2020), juice industry wastewater (Bezirhan Arıkan, et al., 2016), sage (Salvia tomentosa miller), black pepper thyme (Zahter, Thymbra Spicata) and lemon-scented thyme (Thymus zygioides) extracts (Afacan and Kundakcı, 2017), sea lettuce (Ulva lactuca) (Kılıç, 2017), banana peel, pepper stalk and red pine (Özdemir and Ramazanoğlu, 2019) , food waste (Karakuş and Ayhan, 2019), agricultural waste (Samer, et al., 2019), pumpkin (Berkol, 2020), corn husk (Gündüz, 2020) and Posidonia oceanica (Fidan, 2020) was used to produce storage material. In a significant part of these studies, the polymer production process was explained and various properties of the produced polymers were examined. The number of studies (Afacan and Kundakcı, 2017; Gündüz, 2020) in which trials were conducted directly on food is quite limited. In our study, various properties of the produced materials were examined, a direct controlled experiment was carried out on the food, and the results obtained from the gas and temperature sensors were analyzed statistically. Thus, it is thought that our study will contribute to the literature.

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