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# Preparation of Chitosan Biodegradable Polymer Composite: Study of Kinetics and Release Models of NPK Fertilizer in Aquatic Environment

**Somayeh Azizmoradi[1]*, Mostafa Chorom [2], Mohammad Javaherian[3]**

[1]PhD Candidate, Department of Soil Science, Faculty of Agriculture, Shahid Chamran University of Ahvaz, Iran.
[2]Full Professor, Department of Soil Science, Faculty of Agriculture, Shahid Chamran University of Ahvaz, Iran
[3]Assistant Professor, Department of Chemistry, Faculty of Basic Sciences, Shahid Chamran University of Ahvaz, Iran
*Corresponding author

## Abstract

Frankincense fertilizers are a promising way to reduce environmental pollution. In this research, the biodegradable polymer chitosan composite was prepared by in-situ hydrogelation method. The release process of NPK fertilizer, as the most widely used chemical fertilizers used by farmers, was evaluated using this polymer composite. The release of NPK fertilizer was investigated at three different pH levels of 8, 7, and 6 in a period of 720 hours. The ability to release the studied fertilizers and the morphological conditions of the prepared composite were determined by FTIR, XRD, SEM, EDAX. The release mechanism of NPK was investigated by fitting four mathematical models. The results showed that in the zero-order, Higuchi and Krasmir Peppas model, the diffusion coefficient was high in the early stages of fertilizer release.

**Keywords**: Biodegradable composite, slow release fertilizer, chitosan, polysaccharide carrier.

## Introduction

In the last few decades, fertilizer release systems have received more attention in comparison to conventional formwork (Khanam et all., 2025). The function of the main fertilizer release system is the transfer of fertilizer to specific points of the plant where the transfer of fertilizer is done reliably and by adjusting the release mechanism to control the amount of fertilizer and the release time (wang et all., 2025). Various carriers such as polymeric materials, cryohydrates (starch, cellulose, chitosan, xanthan gum, foxglove, dextran, alginate), gels, liposomes, micro-holes were introduced for fertilizer release systems, which pointed out the important advantages of improving product and soil quality, reducing toxicity, and being biodegradable. In the meantime, the use of chitosan will be a promising release system due to its biodegradability and also having important functional groups of hydroxyl and amines (Juntong et all., 2021). The main advantages of this string carrier are the release of fertilizer in a specific location, the embedding of more than one type of fertilizer in the empty spaces of the prepared structure (the porosity of the composite structure), and, depending on the type of nutrient, the increase in its amount within the carrier (Juntong et all., 2021). A review of the conducted research shows that different controlled release systems have been developed in recent years: Deposition on organic/inorganic materials, coating with polymers, encapsulation in matrices, copolymerization through non-flammable bonds (Mandal et all., 2025). A wide range of synthetic polymers were used to make these systems and good results were obtained in terms of long-term release of elements, but they also showed high cost and non-biodegradability (Panpinit et all., 2025). which limits their use. To overcome these drawbacks, formulations based on biopolymers were designed and investigated as an environmentally friendly alternative to those based on synthetic polymers. Polysaccharides are at the forefront of this research because of their natural origin, which gives them biocompatibility, biodegradability, and harmlessness to living organisms (Fertahi, et al., 2021). In addition, they originate from renewable resources and their use helps to conserve non-renewable resources and prevent pollution. In nutrient release systems, fertilizer components are usually mixed with polymer solution and prepared by in situ hydrogel forming method (Fertahi et all., 2021). The morphology of the prepared composite depends on many parameters such as diameter, uniformity, etc (Verma et all., 2025). These parameters are divided into three groups: polymer, device and environment (Verma et all., 2025). The main goal of the present research work is to investigate the release of NPK nutrients from the biodegradable chitosan composite and finally to study the kinetics of the release of loaded nutrients.

## 2. Experimental part:

### 1.2.Necessary materials

Chitosan (biodegradable biopolymer material), carboxylic acids, adipic acid and terephthalic acid are used as crosslinks, thionyl chloride (SOCl2) to connect the crosslinking agent to chitosan polymer, and amino acid cystine to increase the absorption power of cadmium.

### 2.2.Fabrication of chitosan biodegradable polymeric composite:

One tenth gram of chitosan powder (Merck) was dissolved in a mixture of 4.9 ml of water and 35 ml of 1% acetic acid. It is then dissolved at 50°C. Different amounts (in order to maintain the semi-IPN state of the hydrogel, the ratio of amine groups to dicarboxylic acid must be equal to 2) of the crosslinks in question (adipic acid and terephthalic acid separately) in the presence of thinonyl chloride, the hydrogen atom of the hydroxyl agent is lost and replaced by chlorine, and it was prepared for the connection of chitosan strands. When the chlorine atom is removed and the amide bond takes place in the place of the crosslinks between carboxylic acid and chitosan, sesitsen is also added. Then, the desired amount of the desired fertilizers (potassium, phosphorus, nitrogen, respectively) 400, 60, 500 (potassium, phosphorus, nitrogen) (soil test is needed to confirm these values) were dissolved in a 1% ethanol solution in 100 ml of water. (nitrogen source of urea fertilizer, potassium source of potassium sulfate and phosphorus source of triple phosphorus fertilizer). And it was slowly poured into the chitosan solution and vigorously stirred at a temperature of 50 degrees Celsius, then the obtained uniform polymer was subjected to three freeze-thaw cycles (12 hours at -20 degrees Celsius and 4 hours at room temperature until a composite absorbent was formed (Manuela et al., 2019; Meng et al., 2004; Heba et al., 2016).

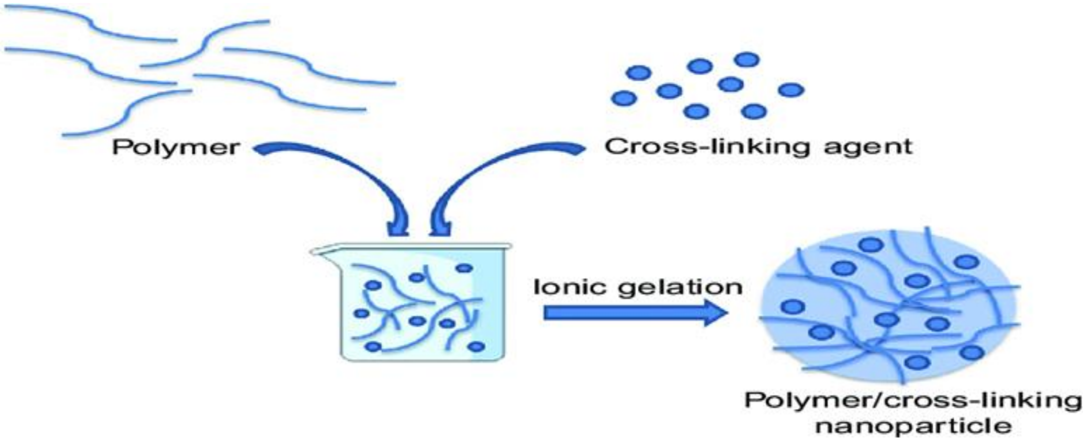


Figure 1- Placement of nutrients in a hydrogel network based on chitosan

### 3.2.Determining the release rate of nutrients from chitosan biodegradation polymeric composite in solution environment:

After completing the freeze-thaw cycle, the Fourier transform infrared spectrometer (FTIR) was used to check the chemical structure and crosslinks, as well as checking the presence of functional groups, determining the type of links in the hydrogel. We used XRD analysis to determine the crystalline phase in the composition and to prove the formation of the composite. Scanning electron microscope (SEM) is used to confirm the creation of biodegradable polymer composite based on chitosan and its morphological characteristics. The dried composite was washed several times with deionized water to remove the superficial material from it. Then it was examined in three solutions with different pHs of 8, 7, and 6 in a time span of 720 hours. Then, nitrogen was evaluated by the Kjeldahl method, phosphorus by the spectrophotometer method, potassium by the film photometer method. The release mechanism is evaluated by examining 5 mathematical models (zero and one order models, Higuchi model Krasmir Peppas model) and then the data obtained from fitting based on the R2 correlation coefficient. The best model will have a higher correlation coefficient and a lower error (SE) (Lan et al., 2008 ., kim et all 2025., Caballero et all., 2025).

4.2. Traditional studies:

The release mechanism is evaluated by examining five mathematical models (zero- and first-order models, Higuchi model, Krasmir Peppas) and then the data obtained from the fitting are evaluated based on the R2 correlation coefficient. The best model will have a higher correlation coefficient and a lower standard error (SE) (Lan et al., 2008).

1.4.2. Zero order model:

The zero order model is the most ideal type of release and it is expected that the group of elements whose level should remain constant in the fertilizer. follow this kinetics, where Qt is the amount of fertilizer dissolved up to time t, k0 is the fertilizer rate constant, and Q0 is the initial amount of fertilizer in the buffer medium, which is usually considered to be zero.

$Q_t = k_0 t + Q_0$

2.4.2. First class model:

The first order model is used to describe the kinetics of absorption or excretion of some fertilizers. Fertilizer release that follows first-order kinetics is described below. in the relation k t is the first order speed constant expressed in time unit. This relationship can be expressed as follows, where Co represents the initial concentration of fertilizer.

$dc/dt = -KC$

3.4.2. Higuchi model:

Higuchi's model is used to express fertilizer release from homogeneous matrices. In this case, the fertilizer release is controlled and limited by the penetration of soluble substances in the matrix, and the release mechanism follows diffusion.

$Q_t = kt$

4.4.2 .Krasmir Peppas

The most comprehensive equation to describe fertilizer release is the well-known semi-empirical equation based on the power law expression developed by Krasmir and colleagues in 1983, where (Mt/M) is the fraction released at time t, n is the release power, and K is the release coefficient. From the slope and intercept of log(Mt/M) versus log(t), kinetic parameters n and K will be calculated. If the value of n is between 2.45 and 2.82, the diffusion and erosion mechanism of the polymer matrix is effective in releasing nutrients. If the value of n is equal to 2.82, the release of food elements is independent of time, and in other cases, no specific mechanism can be mentioned for drug release (Manula et al., 2019).

$m_t / m = kt^n$

3.Discussion and results:

FTIR devices, SEM, XRD, and EDAX devices were used to determine the morphological characteristics of the hydrogel network.

1.3. Examining the results of electron microscopy (SEM):

This microscopic method is used to study the morphology of polymer mixtures and nanocomposites. The magnification of this device can be changed in a wide range from 20 to 300000 times. Tescan VEGA-II scanning electron microscope was used in this project. For this purpose, first the samples are immersed in liquid nitrogen for 2 min and broken. Then their surface is covered with a thin layer of gold with a thickness of A 500-100 so that they have electrical conductivity. The samples are placed in the desired places of the device and the electronic scanning operation of the device begins. Electrons hit the surface of the sample and after returning, an image of the sample is determined. SEM is used to study morphology. Since the porosity of the adsorbent has an important effect on the absorption of heavy metal ions, a scanning electron microscope was used to examine the porosity of nanocomposites grains and their porosity. The sample surface is limited but provides useful information for surface topology with A100 resolution. Figure (2) is related to the SEM of the prepared composite hydrogel, the water coming out of the hydrogel structure causes holes in it. It is clear that with the increase in the number and size of the holes, the amount of absorption also increases. The porous structure of the hydrogel is confirmed according to the shape. Fertilizer particles are embedded in the wall of Manafed. In examining the morphology of the formulation by SEM, there is a relationship between the morphology and the degree of cross-linking (reflected by the acyl chloride linker). In general, increasing the degree of crosslinking creates a denser structure, while increasing the NPK content leads to thicker pore walls. Increasing the NPK content leads to a relatively compact structure with small pores. A porous and homogeneous morphology was observed, with interconnected pores and NPK crystals embedded in the pore walls.

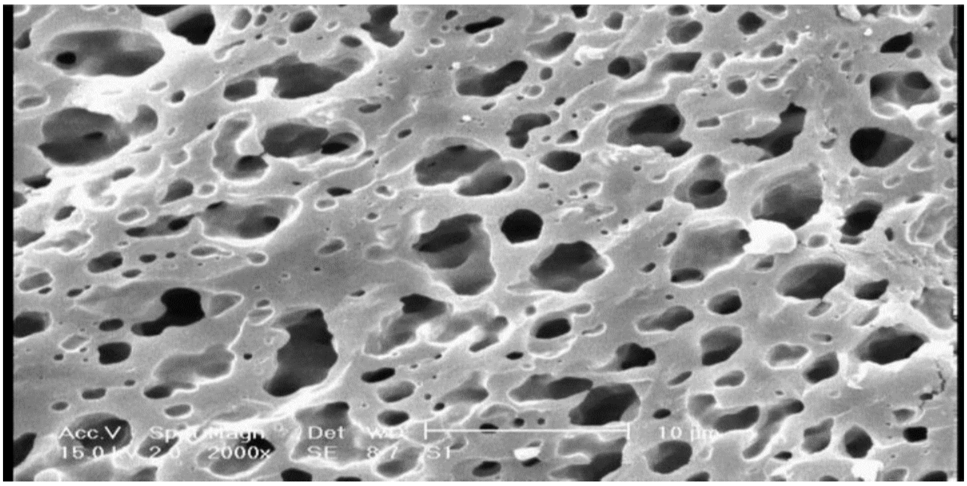
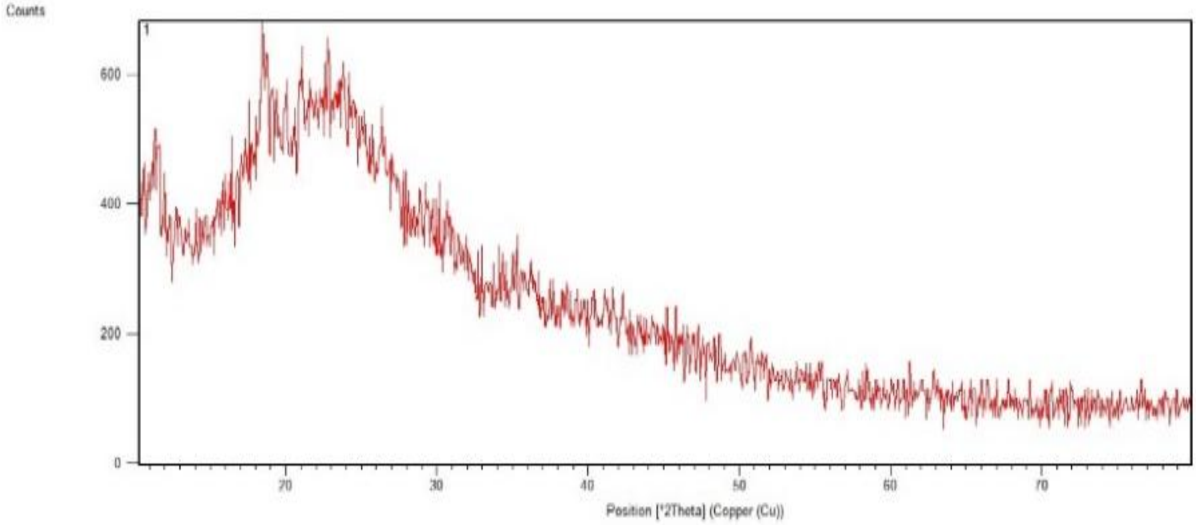


Figure 2. Morphological image of chitosan biodegradable polymer composite

2.3.X.ray diffraction (XRD):

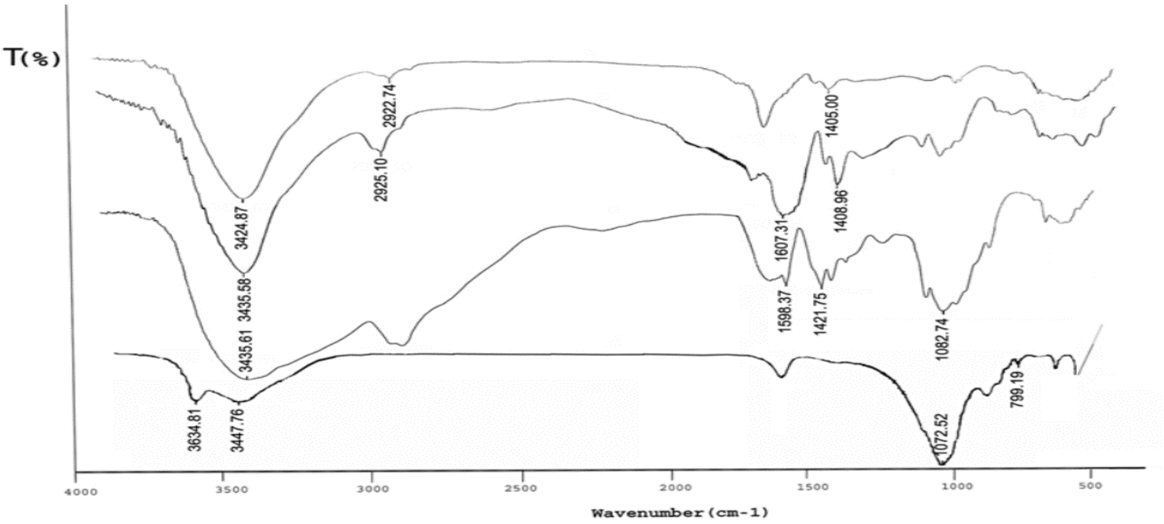
If the sample is in the form of a crystal, the X-rays are attracted to the same shape, as a result, there is no change in the wavelength or phase between the irradiated rays. If the sample is semi-crystalline, the scattering is not isotropic. In such conditions, there will be changes in the wavelength and phase. The peaks located in the region of 10 and 20 A are the XRD spectrum to identify the chitosan phase. Also, the peaks from 2.4 to 29.3 show the fertilizer crystals in the formulation. In predicting the molecular design, cross-linking nodes were confirmed by the presence of broad wide-angle reflections (14 and 20°, respectively) corresponding to intermolecular and interchain distances within the formulation. The existence of balance between the density of imine units and the viscosity of the reaction system shows that the presence of NPK has led to a slight increase in the size of cross-linking nodes. Marian et al also showed during their research that the loading of nutrients (fertilizers) causes a slight increase in the size of cross-linked nodes. The physical forces created between NPK elements and chitosan are involved in the orderly arrangement of imine units. It can be concluded that these forces lead to the stiffening of chitosan chains and thus decrease its mobility, which leads to the self-assembly of newly formed imine units and thus increases hydrogelation. Since NPK elements are in the form of a molecular crystal with hydrogen bonds, their reflections correspond to different lengths of in-plane and out-of-plane hydrogen bonds, and it is likely that these changes affect the physical forces of the NPK chitosan that lead to the formation of NPK crystals enriched with penetration. These results are consistent with those of Popescu et al. 2013.



Finger 3. It shows X-ray scattering in the composite sample

3.3. Spectroscopy: FTIR

FTIR-Bruker EQUINOX 55 Fourier transform infrared spectrometer was used to identify the functional groups in the chemical structure. The sampling method for this device is that some of the nanoparticle is spread on the KBr Pill and then the Pill is placed inside the device and the corresponding spectrum is recorded. In the spectrum of prepared composite hydrogel, the spectrum related to chitosan in the region of 3435 cm1 is related to the stretching vibration of hydroxyl and amine group of the first type, which are covered together. Peak cm1 1598 corresponds to the bending vibration of the amino group, peak cm1 1598, the peak corresponds to the stretching vibration of the bond (amino acids) C-N (Santos, Bacalhau, Pereira, Souza). Peak cm1 1639 is the vibration related to N-H band, the fertilizer particles as tiny crystals are connected to chitosan and hydrogel body by hydrogen bond. The reduction of hydroxyl and amine group bonds indicates physical bonding. The FTIR spectrum of the formula was recorded in order to investigate the hydrogelation mechanism of the system and to have an insight into the encapsulation of NPK elements in them. In the FTIR spectrum, the presence of the characteristic band of the imine bond was given as an intense and sharp band in the range of 1630-1628 and a shift in the broad band from 3000 to 3700, which is attributed to the overlapping stretching bands of O-H and N-H bonds and intra- and intermolecular H bonds. These results are consistent with the results of Iftime et al. 2017.These spectral characteristics were in line with the formation of supramolecular clusters of imine units that played the role of chitosan cross-linkers. In the FTIR spectrum of the formulation, the characteristic bands of the formulation's hydrogel show that despite slight changes in the position and intensity of the components, the maintenance of the hydrogelation mechanism and the occurrence of physical interactions between the two components have been done. The stretching bands of C=O and N-H bonds to NPK elements (1670 cm and 1146 cm/1 cm, respectively) have been moved to higher wave numbers (about 6 cm/1) in the formulation spectra, which indicates their involvement in physical bonds with the hydrogel. No obvious decrease in the intensity of the C=O bond was observed, indicating the absence of condensation reaction with the amine groups of chitosan, as model. As confirmed by the overlapping bands of the hydrogel and the appearance of urea, the separation of the NPK element spectrum from the formulas was applied, when the imine band was clearly observed at 1629 cm, similar to the reference hydrogels. The spectral range of 3000-35000, the formulation is dominated by the vibrational bands of urea amine groups, while the characteristic bands of hydroxyl and methylene units are strongly reduced. Examination of the FTIR data indicated the anchoring of urea to the formulations by physical interactions during hydrogelation. The results of linkage analysis are consistent with those of Santos et al., as well as Haubacalh et al. 2015. Correlating FTIR data with experimental observations, it seems that the occurrence of physical bonds between NPK elements and chitosan favors hydrogelation processes (Iftime et al., 2017).



Finger4.The FTIR spectroscopic diagram of the peaks, from top to bottom, is the hydrogel investigation peak, the composite investigation peak, and chitosan. In comparison, the last peak is the investigation of the hydroxyl group of the SI-O bond in soil and free water.

### 4.3. EDAX analysis:

According to EDAX analysis, which is an add-on in SEM devices, the percentage of fertilizer elements was detected. EDAX analysis is an add-on in SEM devices to detect the percentage of elements in solid samples. EDAX abbreviation of energy dispersive X-ray, sometimes called EDS or EDX. This technique is a method for determining the elemental composition of a sample or part of a sample. EDX is not used alone, but it is a system that is together with the transmission electron microscope (SEM) and is actually a part of this microscope. The output of an EDX analysis is the EDX spectrum. The EDX spectrum is just a graph drawn based on the x-ray energy received from each energy level. Each of the peaks shown in this diagram is specific to one atom and therefore represents only one element. Higher peaks in the spectrum mean higher concentration of the desired element in the sample. The presence of oxygen in the polymer composite is related to the presence of impurities and the physical absorption of moisture from the environment. This is while the increase in the amount of oxygen in the manufactured polymer is related to the creation of oxygen-containing groups. There is a large amount of nitrogen on the polymer. This high amount of nitrogen is due to the type of amine consumed in the consumed amino acids as well as the functional spheres of the chitosan polymer, which has an amine functional group. The presence of sulfur is also related to the amino acid composition of cystine consumed.

Table 1. EDAX sample review

| Elt | K | A% |
|---|---|---|
| C | 0.2870 | 36.08 |
| N | 0.0719 | 14.53 |
| O | 0.3328 | 43.82 |
| P | 0.0215 | 0.41 |
| S | 0.1489 | 2.58 |
| K | 0.0835 | 1.22 |

+

5.3. Fertilizer release kinetics was investigated with four mathematical models:

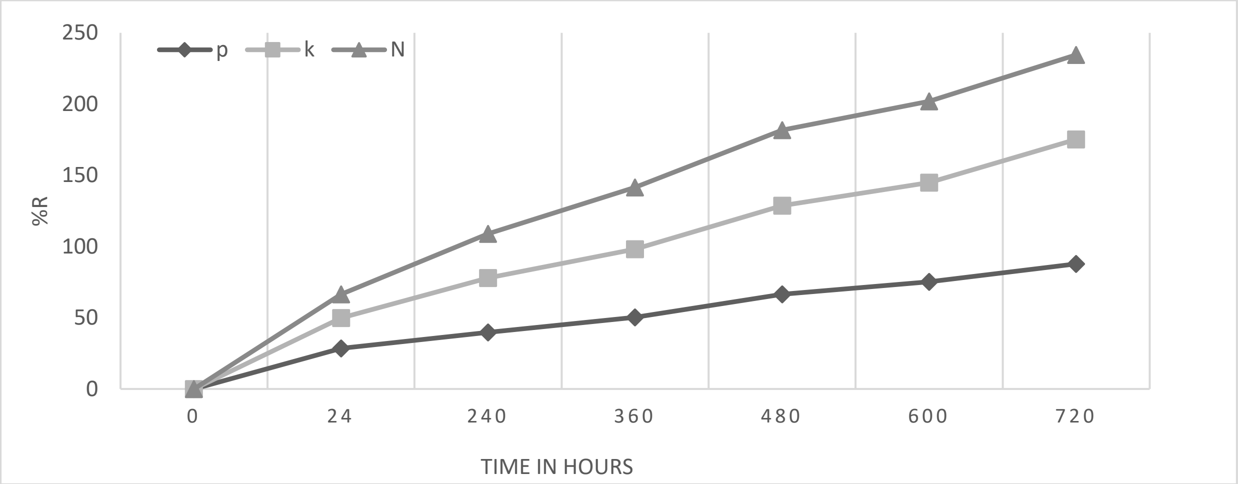
In the zero-order model, Higuchi, Korsmeyer showed a high correlation coefficient in the early stages of fertilizer release. The results of $R^2$ and SE are shown in the tables of the last pages according to three PH=6,7,8. For the studied samples, the excellent fit of the zero-order model shows that the loaded fertilizer has been affected by swelling in the hydrogel in the first twenty-four hours. The good fit of the Higuchi model shows that the spreading process was effective in the fertilizer sector. and it shows the encapsulation of the nutrient part in the body of the hydrogel. The diffusion of fertilizer towards the release environment can be checked with a physical view. There are intermolecular forces between the loaded fertilizer and the body of the hydrogel, which reduces the dissolution of the fertilizer. The presence of interconnectedness in the loaded fertilizer molecules causes a decrease in the speed of their sinking in the direction from the inside to the outside of the hydrogel body due to the presence of a concentration gradient. In addition, the effect of the hydrogel matrix on the retention and release of the fertilizer is confirmed in the Korsmeyer model. In the beginning, it means when water molecules penetrate into the hydrogel network and cause the loaded fertilizer to dissolve. The swelling shows that the movements of the loaded particles inside the hydrogel wall cause pressure in its wall. which definitely causes morphological changes. According to the fit of the models, it can be concluded that the diffusion phenomenon was one of the ways of releasing fertilizer. The existence of this diffusion can be attributed to the presence of larger crystals that are physically anchored to the hydrogel body. These crystals are prone to faster dissolution in the first stages. Considering that the value of n in Korsmeyer equation was 0.2089, here the release model followed the Fickian mechanism.

5.3.1. The next stage of publication compared to the first stage:

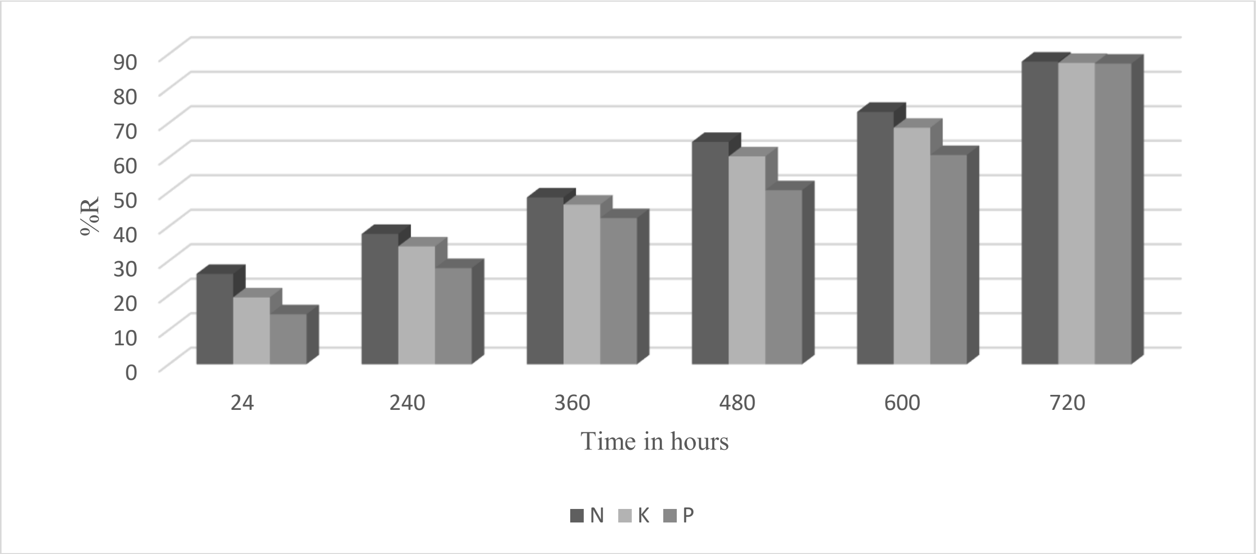
Examining the models showed that in the early stages, some of the loaded particles that are physically anchored to the body of the hydrogel are released during the diffusion phenomenon. According to the fitting of the model of the first stage, in this stage, the amount of loaded particles that remain in the body of the hydrogel plays an important role in its release speed. And in fact, the rate of dissolution of charged particles is more important than the issue of diffusion in this stage. In this stage, due to morphological changes (swelling, cracks in the wall), an accelerated diffusion is provided for the charged molecules. And this diffusion speed itself becomes a driving force for sinking the nutrient part in the direction from the inside to the outside. The remaining particles sink into the body of the hydrogel and their dissolution rate decreases. In fact, it can be said: Wetting the samples allows them to swell. First, according to the SEM images, water has penetrated into the hydrogel through the pores, and then it penetrates the walls of the pores and dissolves the fertilizer particles. In the initial stage, larger crystals that are less connected to the interior of the hydrogel are susceptible to dissolution. After that, the smaller crystals that fit better inside the pores are separated from the hydrogel body and dissolve. It can be concluded that soil moisture will play an important role in the release of elements from this biodegradable polymer composite which is based on chitosan and has a hydrogel state.

Table 2.Synthetic characteristics of nutrient release from chitosan biodegradable polymer composite at pH=6

| | | ph=6 | | | | | |
|---|---|---|---|---|---|---|---|
| | | Time in hours | | | | | |
| Traditional model | Traditional coefficients | 24 | 240 | 360 | 480 | 600 | 720 |
| Zero order model | $R^2$ | 0.9912 | 0.9754 | 0.9703 | 0.9492 | 0.9781 | 0.9642 |
| | SE | 0.59 | 0.58 | 0.48 | 0.44 | 0.39 | 0.28 |
| First order model | $R^2$ | 0.991 | 0.9732 | 0.9704 | 0.9443 | 0.9721 | 0.9619 |
| | SE | 0.52 | 0.41 | 0.38 | 0.35 | 0.32 | 0.21 |
| Higuchi model | $R^2$ | 0.9911 | 0.9731 | 0.9702 | 0.9442 | 0.972 | 0.9607 |
| | SE | 0.21 | 0.24 | 0.18 | 0.11 | 0.13 | 0.1 |
| Krasmir-Peppas model | $R^2$ | 0.9913 | 0.9758 | 0.9709 | 0.9494 | 0.9785 | 0.9643 |
| | SE | 0.68 | 0.52 | 0.48 | 0.5 | 0.43 | 0.62 |



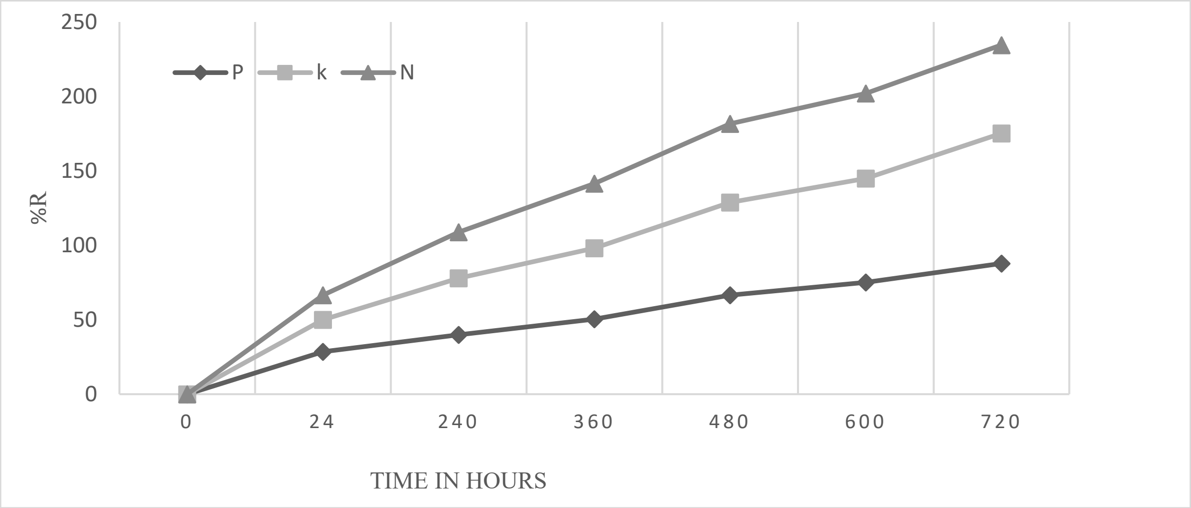
Finger 5.Cumulative graph of release percentage of nutrients from chitosan biodegradable polymer composite at pH=6.



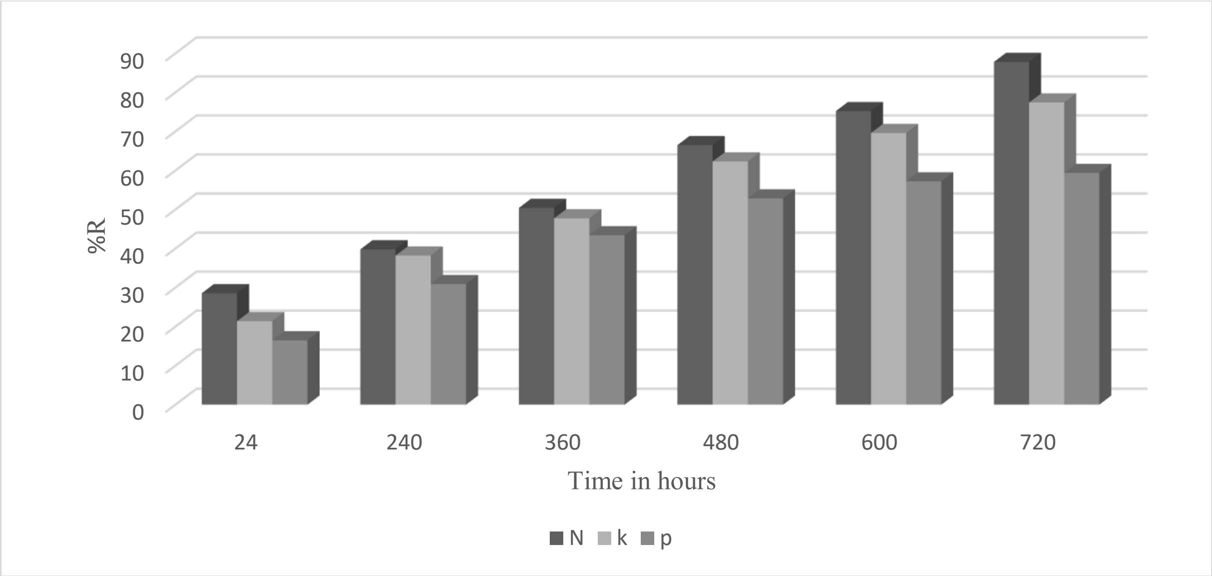
Finger 6. Graph of the percent release of nutrients from chitosan biodegradable polymer composite at pH=6

Table 3. Synthetic characteristics of nutrient release from chitosan biodegradable polymer composite at pH=7

| | | PH=7 | | | | | |
|---|---|---|---|---|---|---|---|
| | | Time in hours | | | | | |
| Traditional model | Traditional coefficients | 24 | 240 | 360 | 480 | 600 | 720 |
| Zero order mode | R2 | 0/9882 | 0.8791 | 0/9814 | 0/951 | 0.9543 | 0.976 |
| | SE | 0.93 | 0.91 | 0.96 | 0.93 | 0.95 | 0.91 |
| First order model | R2 | 0.9874 | 0.8787 | 0.9806 | 0.943 | 0.9538 | 0.9765 |
| | SE | 0.91 | 0.92 | 0.94 | 0.97 | 0.92 | 0.91 |
| Higuchi model | R2 | 0.9872 | 0.8781 | 0.9801 | 0.94 | 0.9526 | 0.9758 |
| | SE | 0.35 | 0.3 | 0.32 | 0.37 | 0.36 | 0.41 |
| Krasmir Peppasmodel | R2 | 0.9884 | 0.8797 | 0.9816 | 0.953 | 0.9548 | 0.9765 |
| | SE | 0.67 | 0.7 | 0.64 | 0.58 | 0.6 | 0.69 |



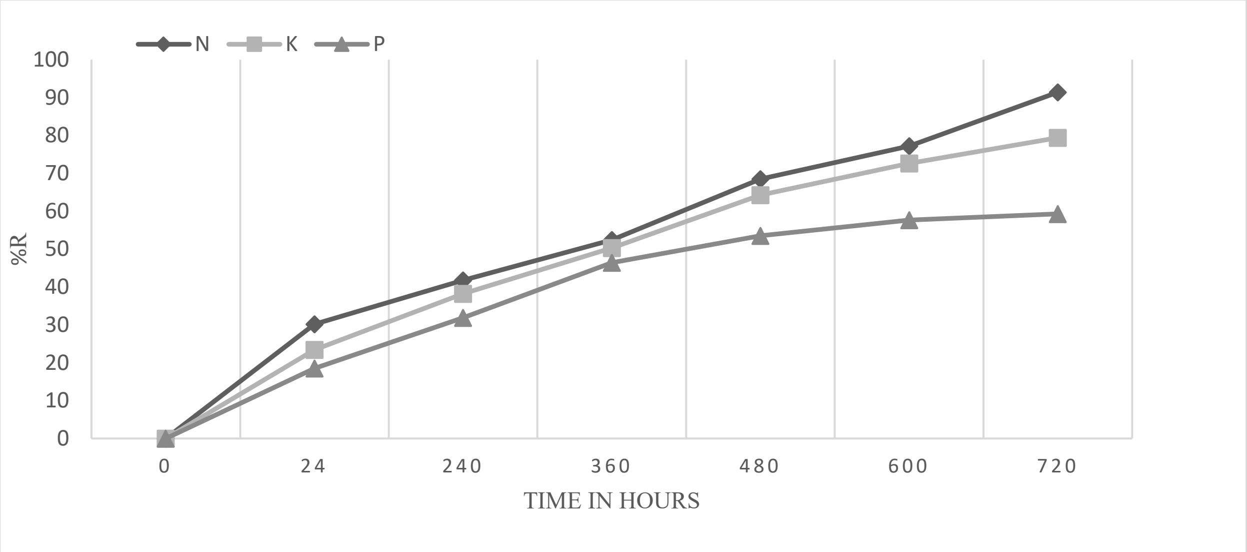
Finger 7. Cumulative graph of release percentage of nutrients from chitosan biodegradable polymer composite at pH=7.



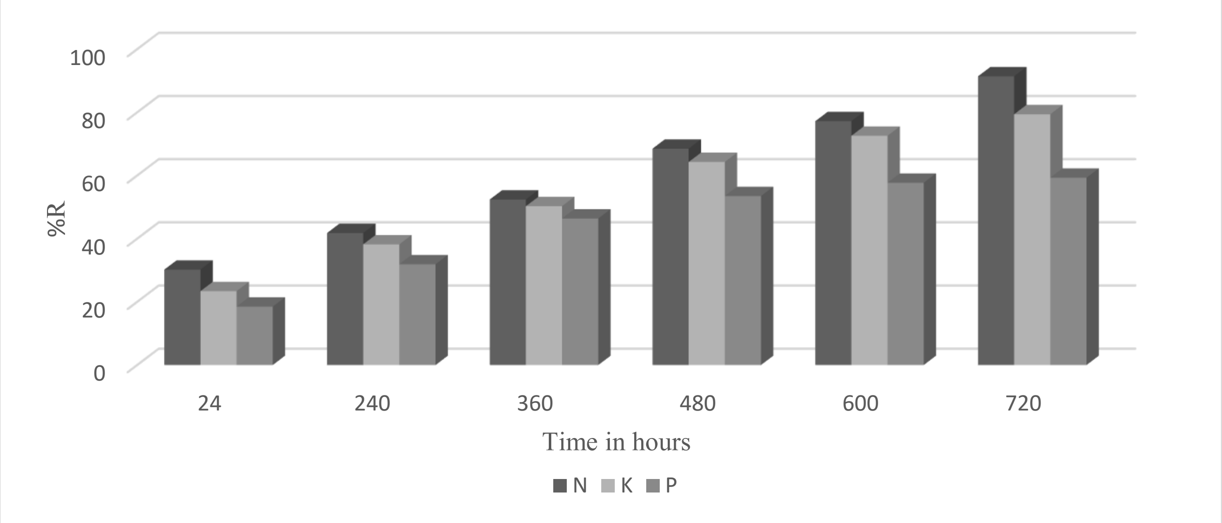
Finger 8. Graph of the percent release of nutrients from chitosan biodegradable polymer composite at pH=7

Table 4. Synthetic characteristics of nutrient release from chitosan biodegradable polymer composite at pH=8

| | | PH=8 | | | | | |
|---|---|---|---|---|---|---|---|
| | | Time in hours | | | | | |
| Traditional model | Traditional coefficients | 24 | 240 | 360 | 480 | 600 | 720 |
| Zero order mode | $R^2$ | 0.9912 | 0.9754 | 0.9703 | 0.9392 | 0.9149 | 0.979 |
| | SE | 0.49 | 0.46 | 0.48 | 0.41 | 0.52 | 0.47 |
| First order model | $R^2$ | 0.9911 | 0.9754 | 0.9702 | 0.939 | 0.915471 | 0.9792 |
| | SE | 0.71 | 0.65 | 0.66 | 0.7 | 0.71 | 0.68 |
| Higuchi model | $R^2$ | 0.9905 | 0.9738 | 0.97 | 0.9388 | 0.9146 | 0.979 |
| | SE | 0.56 | 0.52 | 0.48 | 0.49 | 0.42 | 0.38 |
| Krasmir Peppasmodel | $R^2$ | 0.9913 | 0.9758 | 0.9709 | 0.9394 | 0.9151 | 0.9792 |
| | SE | 0.64 | 0.69 | 0.7 | 0.62 | 0.68 | 0.63 |



Finger9.Cumulative graph of release percentage of nutrients from chitosan biodegradable polymer composite at pH=8.



Finger10.Graph of the percent release of nutrients from chitosan biodegradable polymer composite at pH=8

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