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ABSTRACT

In this work, a smart pH-sensitive carboxymethyl tamarind kernel gum/tragacanth gum/polyacrylamide (CMTKG/TG/PAM) hydrogel for controlled drug delivery of ampicillin was developed. A series of HG were synthesized by varying the composition and optimized based on the swelling ability. The prepared HG were characterized by FTIR, PXRD, and Sem. The cross-linked fractions of the synthesized HG were determined by sol-gel analysis, and the pH sensitivity was established through swelling studies and drug release at pH 7.4 and 1.2. The pH 7.4 demonstrated better swelling and drug release. Further, the kinetic study reveals that the Korsmeyer Peppas model ($R^2 = 0.99$) is found to be the best fit for the drug release study. It was observed that the maximum amount of the drug was released in an alkaline pH. The findings suggest that the synthesized hydrogel (CMTKG/TG/PAM) can be used as a carrier for the oral delivery of ampicillin.

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LIST OF ABBREVIATIONS AND SYMBOLS

AM- Acrylamide

CMTKG- Carboxymethyl Tamarind Kernel Gum

TG- Tragacanth Gum

PAM- Polyacrylamide

KPS- Potassium persulfate

MBA- N, N'-methylene-bis-acrylamide

HG- Hydrogel

g- Grams

mg- Milligrams

rpm- Rounds per minute

DL- Drug Loading

DR- Drug Release

ml- Milliliter

FTIR- Fourier Transform Infrared Spectroscopy

XRD- X-ray Diffraction

SEM- Scanning Electron Microscope

UV- Vis- Ultraviolet-visible Spectroscopy

R^2- Regression Coefficient

%- Percentage

λ_{max}- Maximum Wavelength

w_d- Weight of dried hydrogel

w_s- Weight of swollen hydrogel

SR- Swelling Ratio

DW- Distilled Water

19 PEG- Polyethylene Glycol

PVA- Polyvinyl Alcohol

PAA- Polyacrylic acid

CHAPTER 1

INTRODUCTION

Introduction

Colloidal gel was first referred to as "Hydrogel" in 1894. In 1960, Wichterle and Lim discovered the first use of hydrogel in the biomedical industry. [1].This term “Hydrogel” is made of two words i.e. “Hydro” means water and “gel” means a thick substance which is between a solid and a liquid. Hence, we can say Hydrogel is a gel in which the liquid component is the water.[2]

A three-dimensional network of cross-linked polymers called hydrogel has the capacity to absorb enormous volumes of water. It is composed of both solid and liquid phase, therefore it shows both the properties of solid as well as the liquid.[3] HG have wide applications in drug delivery, tissue engineering, wound dressing, contact lenses, agriculture and biosensors. HG are soft and flexible in nature due to their high-water content, three-dimensional polymeric network etc. There are various properties which result from their soft and flexible nature like elasticity, ability to bend or compress, comfort in contact with tissues and good shape adaptability.

1.1 Types of HG

There are various factors which can be used to categorize HG. These factors can be source, polymer composition, cross-linked method, electrical charge, response to stimuli etc. ‘Peppas’ identified three broad categories, ranking them accordingly,

(1) Their preparation process;

(2) Ionic charge;

(3) A certain physical structure;

On the basis of their polymer composition their are three categories of polymers.

I) **Homo-polymeric HG**

These are formed from a single type of monomer unit. Polyacrylamide is an example of this type of hydrogel.

II) **Copolymeric HG**

These contain two or more different monomer units, with at least one being hydrophilic. Poly (acrylic acid-co-acrylamide) is an example of this type of hydrogel.

III) **Interpenetrating Polymer Network (IPN) HG**

These consist of two independent networks which are not covalently bonded but physically intertwined. They have high mechanical strength, enhanced stability and better elasticity.

21 According to "Peppas," ionic charges can be used to create three different kinds of HG. The e. ampholytic, anionic, cationic, and neutral. Anionic HG have negatively charged groups, cationic HG have positively charged groups, and ampholytic HG have both positively and negatively charged groups, giving them a dual nature. [4] HG can be divided into three main groups based on their physical structure. i.e. crystalline HG having dense ordered macromolecular chains and electrostatic interactions, amorphous HG containing unordered macromolecular chains arranged in a random manner, and semi- crystalline HG are composed of both amorphous and crystalline phases.

HG can also be classified on the basis of their cross-linking method:

I) **Physically Cross-Linked HG**

2 These HG exhibit non-covalent interactions, including hydrogen bonding, ionic interactions, or crystallization.

II) **Chemically Cross-Linked HG**

These have covalent bonds between the polymeric chains due to which they have permanent structure and better stability.[5]

1.2 Composition of HG

The composition of HG determines their swelling characteristics, porosity, biodegradability and mechanical properties. A hydrogel typically consists of a polymeric matrix, cross- linking agent and water.[6] Natural polymers, synthetic polymers or combinations of both may be used as the network- forming materials. These are classified into different classes like

2 polysaccharides such as chitosan, chitin, cellulose, starch, alginate, gums, and carrageenan, biological polymers as nucleic acid and DNA, polyamides such as collagen, polyphenols include lignin, inorganic polyesters like polyphosphazene, organic polyesters.[7] The cross-linking density directly influences the ability of the network to absorb water. Additional components such as nanoparticles, drugs, proteins and bioactive molecules may be incorporated to obtain multifunctional systems. Careful optimization of composition is

essential for achieving desired performance in pharmaceutical and biomedical applications. [8,9]

1.3 Natural Polymeric HG

Natural HG are polymeric networks which are derived from the naturally occurring biomaterials which have the ability to retain a significant amount of water. They are composed of hydrophilic polymers containing functional groups such as hydroxyl, carboxyl, amino, and sulfate groups, which facilitate water absorption and swelling. Polysaccharides are the most commonly used natural polymers which consist of long chains of carbohydrate molecules linked by glycosidic bonds and possess excellent water- binding capacity. Some other examples also include guar gum and tragacanth gum. These types of polymers have excellent biocompatibility, biodegradability and low toxicity. These can be prepared via physical cross-linking methods as well as the chemical cross- linking methods. They also have wide applications in drug delivery systems, tissue engineering, wound dressing, agriculture and environmental applications. [10,11]

1.4 Synthetic Polymeric HG

Synthetic polymer HG are three dimensional cross- linked polymers prepared from the polymers which are chemically synthesized. These HG exhibit controlled physical, chemical and mechanical properties due to which they become suitable for advanced biomedical, pharmaceutical, industrial and environmental applications. They have high water absorption capacity, controlled mechanical properties, and structural stability. There are various types of synthetic polymer HG like Polyvinyl Alcohol (PVA) HG (high mechanical strength, good biocompatibility, and chemical resistance), Polyethylene Glycol (PEG) HG, Polyacrylamide (PAM) HG, poly (N-isopropyl acrylamide) (PNIPAM), Poly (acrylic acid) (PAA) etc. These have wide applications in drug delivery systems, contact lenses, wound healing, biosensors, and agriculture. [12,13]

Natural Polymers with Synthetic Polymer HG

Hybrid HG formed by combining natural and synthetic polymers have emerged as a promising strategy for overcoming the limitations of individual systems. Natural polymers contribute biodegradability and biocompatibility, whereas synthetic polymers enhance mechanical strength and structural stability. Examples include guar gum-polyacrylamide, tragacanth gum-polyacrylamide and chitosan-polyvinyl alcohol systems. Such HG often

demonstrate superior swelling behaviour, improved drug loading efficiency and controlled release characteristics. Hybrid systems are increasingly being explored for biomedical, environmental and agricultural applications. [14,15]

HG Based on Poly (Acrylamide) Derivatives

The HG based on Poly(acrylamide) derivatives are widely used in biomedical, pharmaceutical, agricultural, and environmental applications. These HG are formed from acrylamide via polymerization and cross-linking reactions that create a three -dimensional hydrophilic network due to which it is capable of absorbing large amounts of water.[16].Basically, PAM is synthesized from the monomer unit acrylamide through free radical polymerization. The backbone is the carbon-carbon polymer chain along with functional groups. The derivatives are prepared through chemical modification or copolymerization which enhances the responsiveness, swelling capacity, biodegradability, and mechanical properties of the hydrogel. The HG are capable of retaining large amounts of water due to the presence of the hydrophilic amide groups. These are transparent which makes them a good fit for the applications such as contact lenses and electrophoresis etc. They are stimuli responsive like used to respond to pH, temperature, and electric fields etc. It can also be synthesized through gamma radiation or electron beam irradiation without chemical initiators. Continuous advancement in polymer chemistry is expected to further expand the applications and effectiveness of polyacrylamide derivative HG in modern scientific and biomedical research.

1.5 CMTKG //Carboxymethyl Tamarind Kernel Gum.

Carboxymethyl Tamarind Kernel Gum is a derivative of the natural polysaccharide tamarind kernel gum, which is sourced from the seeds of the tamarind tree, Tamarindus indica. CMTKG is produced by reacting tamarind kernel gum with monochloroacetic acid in an alkaline environment, leading to the replacement of some hydroxyl groups in the polymer with carboxymethyl groups. The extent to which hydroxyl groups are substituted significantly influences the final characteristics of the polymer. Addition of carboxymethyl group into the polymer backbone enhances its physiochemical properties. It has various properties like biocompatibility, biodegradability, non- toxicity, and fi- forming ability. The modification improves the water solubility, hydration capacity, and swelling behaviour of tamarind kernel gum. Therefore, CMTKG can absorb more amount of water and form stable gel hydrogel structures. The carboxymethyl group also increases the number of hydrophilic sites in the

polymer which promotes greater interaction with the water molecules. It also enhances the mechanical strength and controlled swelling properties which provide active sites for cross-linking reactions. It has various application in certain fields i.e.pharmaceutical delivery mechanisms, materials for wound repair, scaffolding for tissue engineering and water treatment technologies. It has great advantages like derived from renewable natural resources, economical and readily available, environmentally friendly, easy chemical modification, and good compatibility with other polymers.

1.6 Tragacanth Gum

Tragacanth gum is a natural exudate obtained from Astragalus species and consists primarily of tragacanthin and bassorin fractions. Tragacanthin is the water-soluble fraction of the gum which forms colloidal solution in water, improves emulsifying properties, and contributes to viscosity. Bassorin is the water insoluble fraction but swellable which absorbs water and swells rapidly, provides thickening ability and is responsible for gel formation. Tragacanth gum consists of various monosaccharide components like Galacturonic acid, Galactose, Xylose, Fucose, Arabinose etc. It is prepared in several steps like first dried exudate is being collected which is followed by cleaning and removal of impurities and after grinding into powder form, it is purified and dried. It is then used in the formation of hydrogel formation, its hydroxyl and carboxyl group molecules interact with water molecule and cross-linking agents to form a three-dimensional polymeric network. It has various physical and chemical properties like high water absorption, viscosity enhancement, gel forming ability, biocompatibility, biodegradability, and stability. Tragacanth gum is modified to increase its mechanical strength and functional properties. It has wide applications in pharmaceuticals in tablet binder, suspending agent, emulsifier, and controlled drug release material. It is also used in the biomedical field in wound dressings, and drug delivery systems. Cosmetic industry and Food industries also show wide applications of tragacanth gum as thickening agent, stabilizer, texture enhancer etc. One of the major advantages of tragacanth gum is its natural origin and biodegradability which makes it environment friendly compared to many synthetic polymers. It is also stable over a range of pH and temperature. The variation in composition depends on the plant source and harvesting conditions. Tragacanth gum needs to be stored and processed properly in order to prevent microbial contamination. Overall, Tragacanth gum is an important natural polymer with various applications in many fields. Its various physical and chemical properties make it an important material for developing sustainable and advanced functional systems.

CHAPTER 2

MATERIALS AND SYNTHESIS

2. MATERIALS AND SYNTHESIS

2.1 Materials

All chemicals, solvents, and reagents employed in this study were used as received, with distilled water (DW) adjusted to pH 7 serving as the medium for the preparation of solutions. Buffer systems with pH values of 1.2 and 7.2 were freshly prepared in the laboratory for experimental use. Tragacanth gum (TG) was produced from CDH Fine Chemicals Limited, India. CMTKG with a degree of substitution of 0.2, was supplied by Hindustan Gum and Chemicals Limited, India. Acrylamide (AM), potassium persulfate (KPS), and N,N'-methylene-bis-acrylamide (MBA) were obtained from Merch, Germany, and used without further purification. Ampicillin, used as the model drug, was provided by M/S Unicure (India).

2.2 Synthesis of CMTKG/TG/PAM hydrogel and drug-loaded CMTKG/TG/PAM hydrogel

Table 1 depicts the formulation of the synthesized hydrogel. Initially, 0.3g of CMTKG powder was added to a beaker containing 10mL of distilled water, the mixture was stirred for 30 minutes at 450 rpm until a homogeneous mixture was formed. Meanwhile, in another beaker containing 10 ml od distilled water, 0.15g of TG powder was stirred at 450 rpm for 30 minutes until it became uniform. These two mixtures were then combined, and 0.6g of AM was added, and the mixture was blended for about 10 minutes at 450 rpm. Next, 0.06g of KPS (initiator) and 0.06g of MBA (crosslinker) were added to this mixture and stirred for 1 hour. Then 50 mg of ampicillin was added, and the solution was stirred until a homogeneous solution was achieved. Further, transferred to the test tube and heated in a water bath at 60°C. After an hour, the test tube was removed, and the product was retrieved by smashing the tube and cutting the material into circular rings. Finally, the product was dried in an oven at 60°C until the HG reached a constant weight, presented in Figure 1.

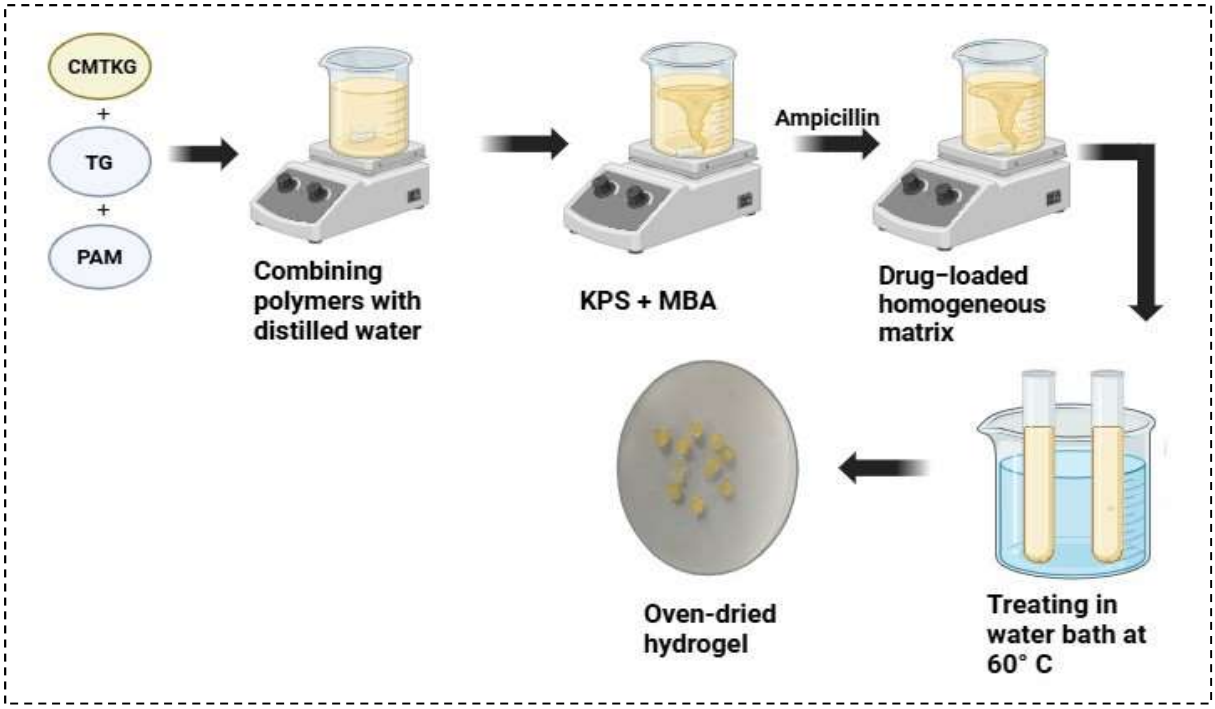

Figure 1. Free radical polymerization of drug impregnated CMTKG/TG/PAM hydrogel

Table 1. Hydrogel preparation and Percentage of swelling

Sample Code	CMTKG (g)	TG (g)	AM (g)	KPS (g)	MBA (g)	Swelling Ratio (%) pH 7.4	Swelling Ratio (%) pH 1.2
J-0	0.3	0	0.7	0.07	0.05	1190	880
J-1	0.3	0.05	0.7	0.07	0.05	1230	917
J-2	0.3	0.1	0.7	0.07	0.05	1263	928
J-3	0.3	**0.15**	0.7	0.07	0.05	1298	946
J-4	0.3	0.2	0.7	0.07	0.05	1286	932
J-5	**0.3**	**0.15**	**0.7**	0.08	0.05	1275	937
J-6	0.3	0.15	0.7	**0.06**	0.05	1316	968
J-7	0.3	0.15	0.7	0.05	0.05	1302	953
J-8	0.3	0.15	0.7	0.03	0.05	1277	929
J-9	**0.3**	**0.15**	**0.7**	**0.06**	**0.06**	**1379**	**984**
J-10	0.3	0.15	0.7	0.06	0.07	1345	972

J-11	0.3	0.15	0.7	0.06	0.030	1267	898
J-12	0.3	0.15	0.7	0.06	0.025	1227	878

CHAPTER 3

EXPERIMENTAL SECTIONS

3. EXPERIMENTAL SECTION

3.1 Swelling Study

The Swelling behaviour of the HG was studied to assess their response under conditions, namely physiological (pH 7.4) and gastric (pH 1.2) environments. This study was conducted using a gravimetric method over a 24-hour period. Hydrogel discs of approximately 0.1g were dipped in 100 mL of buffer solutions at both pH levels. At regular intervals, the samples were taken out gently and blotted dry with filter paper to clean extra liquid, weighed, and then returned to the buffer for continued measurement.

The swelling ratio (%SR) was determined by e:

$$\text{Swelling Ratio(\%SR)} = \frac{(w_s - w_d)}{w_s} \times 100$$

Each experiment was performed 3 times to ensure reliability. Ther results were expressed as mean ± standard deviation and error bars were used to indicate deviation among samples.

3.2 Sol-Gel Fraction

The sol-gel part of the HG we made was looked at using a way to separate the parts that can be dissolved in water and the parts that cannot. First, we dried the hydrogel discs until they stopped losing weight, which we called weight one. Then we put these dried discs in clean water and put them in a warm oven at 100℃ for 5 hours. This helped get rid of the polymer that was not connected properly. After that the HG were wet so we dried them again at a temperature 60℃ until they stopped losing weight again which we called weight two. We did this to find out the sol-gel fraction of the HG.

The sol gel fractions were calculated using Equations:

$$\text{Sol fraction} = \frac{w_1 - w_2}{w_1} \times 100$$

$$\text{Gel fraction} = 100 - \text{Sol fraction}$$

3.3 Percentage Drug Loading (%DL)

Drug Loading was carried out using formulation J-9, as this hydrogel showed the highest swelling among all tested samples. The same synthesis method was followed for preparing the ampicillin sodium-loaded CMTKG/TG/PAM hydrogel, with one small change: 40 mg of ampicillin sodium was added to the solution together with MBA and KPS. All other steps remained the same. For testing, the pre-weighed drug-entrapped HG were placed in 100mL

of buffer solution (pH 7.4) for 24 hr. From this solution, 2 ml sample was taken, and the absorbance was measured at λ_{max} 218 nm using a UV-Vis spectrophotometer (Model: UV-Vis 1900i). After extraction, the HG were removed, air-dried, and then oven at 60degrees C.[34]

The percentage of drug loading and entrapment efficiency was calculated using Equation:

$$\text{DL } (\%) = \frac{Amount\ of\ drug\ in\ hydrogel}{Weight\ of\ hydrogel\ disc} \times 100$$

3.4 Percentage Drug Release (%DR)

Ampicillin release from CMTKG/TG/PAM HG in PBS buffer solutions having pH values of 7.5 and 1.2 was performed. During each experiment, 0.1 g of drug loaded hydrogel was dissolved in 100mL of buffer solution. The solutions were kept in an incubator shaker machine at 110 rpm at a temperature of 37degrees C. From the solutions, samples were collected at regular intervals by withdrawing 2mL volume and adding fresh buffer of same volume in solution to maintain the same volume for 24 h. After this, there was np more release of drug. For measurement of release of drug, Beer's Lambert's law was used at λmax= 218 nm to calculate the concentration of released drug. A standard graph was plotted by UV- Visible spectrometry. Then Cumulative drug release (CDR) was determined by: [35]

$$\text{CDR } (\%) = \frac{Amount\ of\ drug\ released}{Total\ drug\ loaded} \times 100$$

3.5 Kinetic Models of Drug Release

The experimental release data were fitted to different kinetic models, including zero-order, first-order, Korsmeyer-Peppas, and Higuchi models, as shown in Table 2. The suitability of each model was assessed by comparing the coefficient of determination (R^2) values. The model with an R^2 value closest to 1 was considered to provide the best fit for describing the drug release behaviour.

Table 2. Kinetic models for drug release

Kinetic Models	**Equation**	**Mechanism**	**Ref.**
Zero Order	$M_t = M_\infty + k_0 t$	The amount of drug released is independent of the rate	34
First Order	$loglog\ M_t = loglog\ M_\infty + \frac{kt}{2.303}$ $k = rate\ constant$	The release rate is linearly dependent on the amount of the drug	35
Higuchi	$F = \frac{M_t}{M_\infty} = k_H t^{\frac{1}{2}}$ $k_H = kinetic\ constant$	It follows the Fickian diffusion method and can explain the hydrophilic and hydrophobic release of drugs	36
Korsemeyer-Peppas	$F = \frac{M_t}{M_\infty} = kt^n$ k = kinetic constant, n= diffusion	It depends on the diffusion exponent value and stated three conditions in the mechanism 1) if (n = 0.5), Fickian Diffusion 2) if (0.89 > n > 0.5), Non -Fickian Diffusion 3) if (n > 0.89), Case II transport	37

CHAPTER 4

RESULT AND DISCUSSION

4.1 Overview of CMTKG/TG/PAM hydrogel and ampicillin-loaded CMTKG/TG/PAM hydrogel formation

As shown in table 1 we made kinds of hydrogel formulations. We used a radical polymerization method.We used gum, potassium persulfate and N N'-methylene-bis-acrylamide in different amounts.When we heated it to around 60 degrees the potassium persulfate broke down. Formed sulfate radicals.These sulfate radicals then attacked the acrylamide. Created active sites.These active sites helped form polyacrylamide chains.At that time the sulfate radicals took hydrogen atoms from the carboxymethyl tamarind kernel gum and the tragacanth gum.This action made sites on the natural polymer backbones, which are the carboxymethyl tamarind kernel gum and tragacanth gum.The N, N'-methylene-acrylamide acted like a bridge.It connected the radicals on the carboxymethyl tamarind kernel gum, tragacanth gum and polyacrylamide chains.This helped create a three- hydrogel network.The chemical reactions started, grew and then stopped. Also as we can see in Figure 2 the hydrogen bonds between ampicillin sodium and polyacrylamide helped keep the drug stable, inside the hydrogel. The hydrogel is made from carboxymethyl tamarind kernel gum, tragacanth gum and polyacrylamide. The hydrogel has ampicillin sodium loaded in it.This is how we created a hydrogel loaded with ampicillin sodium. The hydrogel is made using carboxymethyl tamarind kernel gum, tragacanth gum and polyacrylamide.

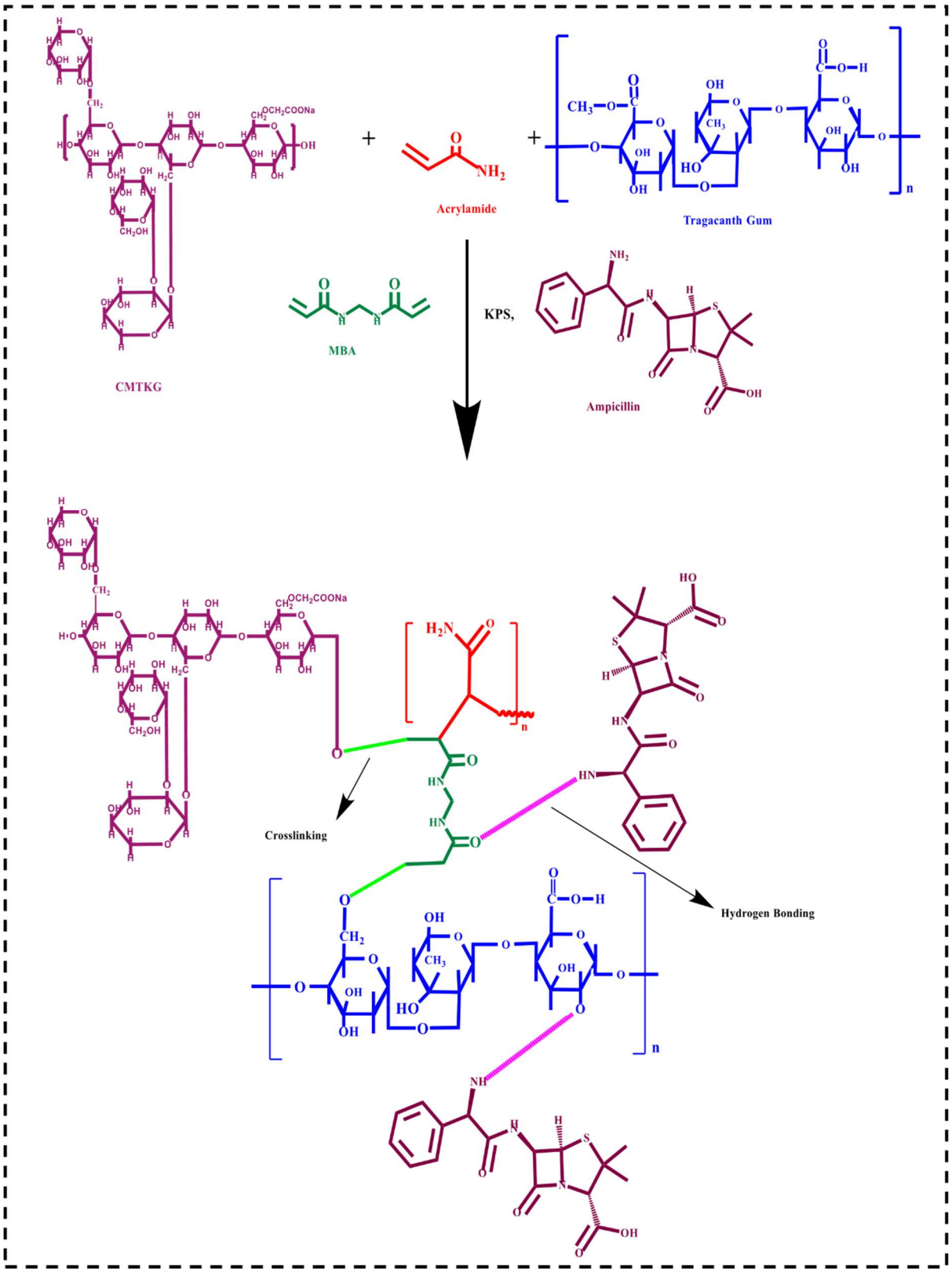

Figure 2. Proposed reaction mechanism of drug loaded CMTKG/TG/PAM composite hydrogel formation

4.2 CHARACTIZATION TECHNIQUES

4.2.1 FTIR

The scientists used FTIR spectroscopy to look at what's going on inside the hydrogel matrix and to figure out what the hydrogel matrix is made of. They observed an absorption band at 3195 cm-1, which is associated with the hydrogel network's O-H and N-H group vibrations. This large absorption band is most likely caused by the hydrogel matrix's high hydroxyl group content, which indicates that the hydrogel matrix's cross-linked polymer network truly enjoys water. The presence of carboxyl groups in the hydrogel matrix is indicated by the C-H stretching peak at 2970 cm-1 and the C=O stretching peak at 1651 cm-1. These hydrogel matrix carboxyl groups may have originated from amide or carboxylate groups in the hydrogel matrix's polymer backbone. The hydrogel matrix's backbone structure remained unchanged when the drug was added because the FTIR spectra of the hydrogel matrix containing the drug and the hydrogel matrix without the drug were identical. The wavenumber region between 1700 and 500 cm-1 did, however, undergo some changes. especially the surrounding peaks.

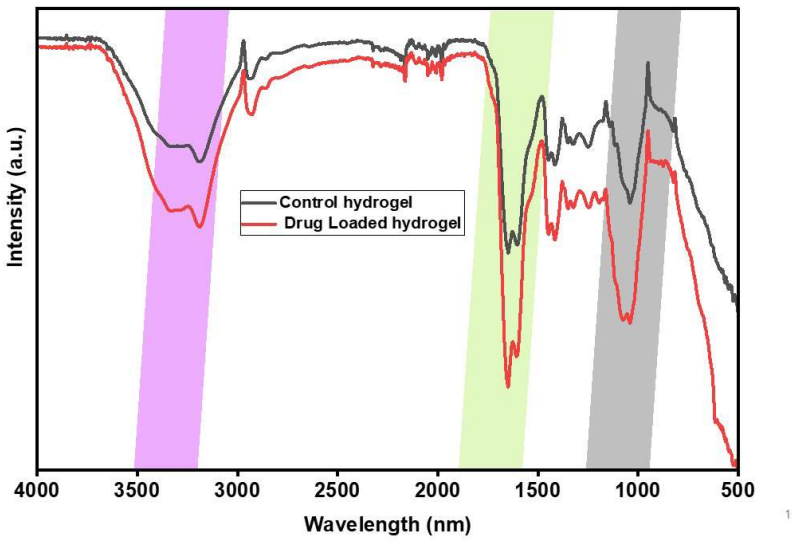

Figure 3. FTIR of CMTKG/TG/PAM hydrogel and drug-loaded CMTKG/TG/PAM hydrogel composites

4.2.2 XRD

X-ray diffraction was done to check how crystalline the hydrogel network was.The diffraction patterns were recorded for ampicillin, the control hydrogel and the drug-loaded hydrogel as you can see in the figure. The control hydrogel showed a peak range between 15˚ and 30˚. There were no sharp peaks. This confirmed that the control hydrogel was amorphous.The drug-loaded hydrogel showed some changes, in peak positions. We saw peaks at 22.4˚, 31.5˚, 32.82˚ and 44.76˚.These peaks came from the crystalline drug. This means the drug was successfully added to the hydrogel.The drug still had its crystalline structure when it was mixed with the hydrogel.The hydrogel network was amorphous. The drug kept its crystalline form.The drug-loaded hydrogel had these peaks because of the crystalline drug.

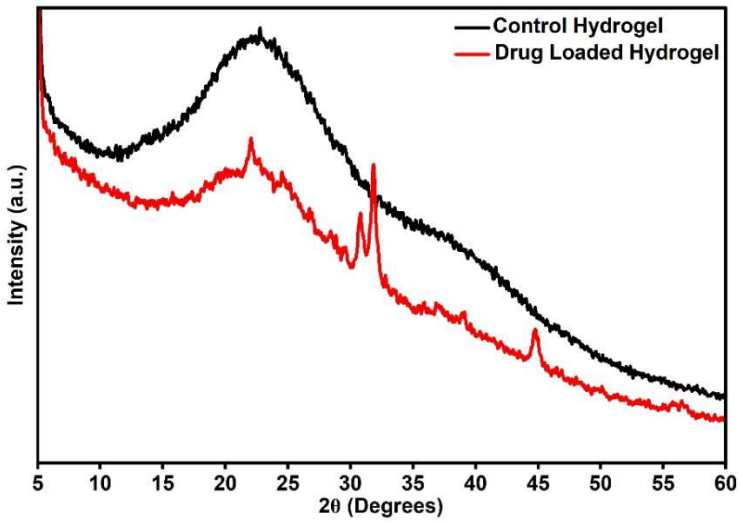

Figure 4. The plot of PXRD of CMTKG/TG/PAM and drug-loaded CMTKG/TG/PAM hydrogel

4.2.3 SEM

Figure 5 shows what the CMTKG/TG/PAM hydrogel looks like under a microscope.We also looked at the CMTKG/TG/PAM hydrogel when it had sodium in it.The hydrogel without any medicine had a surface that was not very smooth and had a lot of holes.This made it easy for the ampicillin sodium crystals to get stuck inside the hydrogel.When we looked at the hydrogel with the medicine the surface was smoother and did not have as many holes.This change happened because the holes, in the hydrogel got filled up with the medicine that was trapped inside the CMTKG/TG/PAM hydrogel.

Figure 5. The plot of SEM of (a) CMTKG/TG/PAM and (b) drug-loaded CMTKG/TG/PAM hydrogel composites

4.3 Effect of swelling ratio

4.3.1 Effect of Biopolymer- Tragacanth Gum

When I changed the amount of gum or tragacanth gum between 0.05g and 0.20g I noticed the HG started to swell more.The swelling ratio went up from 0.05 to 0.15.This makes sense because tragacanth gum loves water, which helps it take in fluid.You can see this in Figure 6a.If I added much tragacanth gum, up to 2g the swelling ratio started to go down a bit.At these levels the solution got really thick and hard to mix.This made it tough to form HG.I also looked at how pH affects the HG.The HG swelled more in alkaline conditions than, in ones.At 7.4 the polymer network can expand easily.This allows more water to get in and stay in.At pH 1.2 it's harder for water to penetrate and be retained.The tragacanth gum and HG worked better in alkaline conditions.The tragacanth gum helped the HG swell more.The HG swelled more with gum.The swelling ratio increased with gum.

4.3.2 Effect of initiator – KPS

When the amount of KPS was changed it was clear how it affected the swelling of the hydrogel. The crosslinker MBA was kept the same. The hydrogel swelled more as the KPS concentration went up to 35 mg. This happened at both 7.4 and pH 1.2. The reason the hydrogel swelled more at KPS concentrations is that it made a lot of free radicals. These radicals helped the polymerization process. Created a strong crosslinked network. If there was not KPS it did not make, as many radicals. This resulted in a network. If there was too much KPS it caused other reactions to happen. This included the formation of oligomers and

radical interactions. These reactions stopped the chain from growing and damaged the hydrogel network.

4.3.3 Effect of cross-linker – MBA

The swelling behaviour of the HG was strongly influenced by that amount of crosslinker (MBA), as shown in Figure 6c. The formulation containing 0.06g MBA produced the highest swelling ratio in both pH conditions. When the MBA concentration was increased beyond this level, the swelling ratio declined slightly. This reduction can be explained by the denser crosslinking, which tightened the network, reduced pore size, and limited the movement of fluid into the matrix.

In contrast, when the MBA content was lower than the optimum, the network did not form with the sufficient strength. The result was an irregularly crosslinked structure that behaved more like a soft gel than a stable hydrogel.

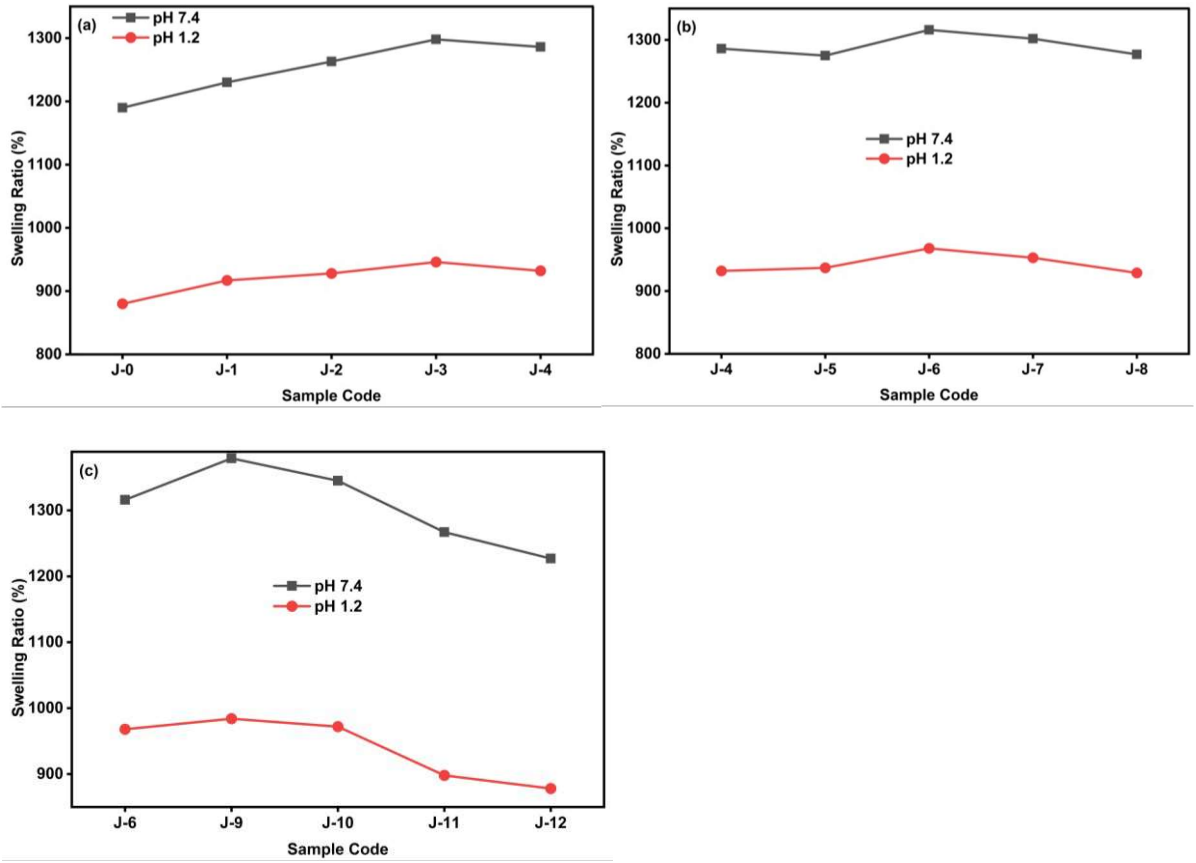

Figure 6. plots for the impact of (a) biopolymer - TG, (b) initiator, (c) cross-linker

4.3.4 Effect of swelling ratio in different pH solutions

The swelling behaviour of the HG was clearly influenced by pH as shown in Figure 7. All samples swelled more at pH 7.4 than at pH 1.2. This response can be explained by the chemistry of the carboxyl groups:in alkaline conditions they lose protons and become ionized. The resulting charges repel each other. Force the polymer chains apart which expands the network.In conditions ionization is suppressed so the chains stay closer together and swelling is minimal.Overallthe study confirms that HG are strongly pH-responsive.Their ability to expand and absorb fluid varies significantly depending on the surrounding medium and pH.The HG respond well to changes.The study is about HG and their swelling behaviour.It shows that HG can expand and absorb fluid in ways, at different pH levels.The HG swelling behaviour is affected by pH.

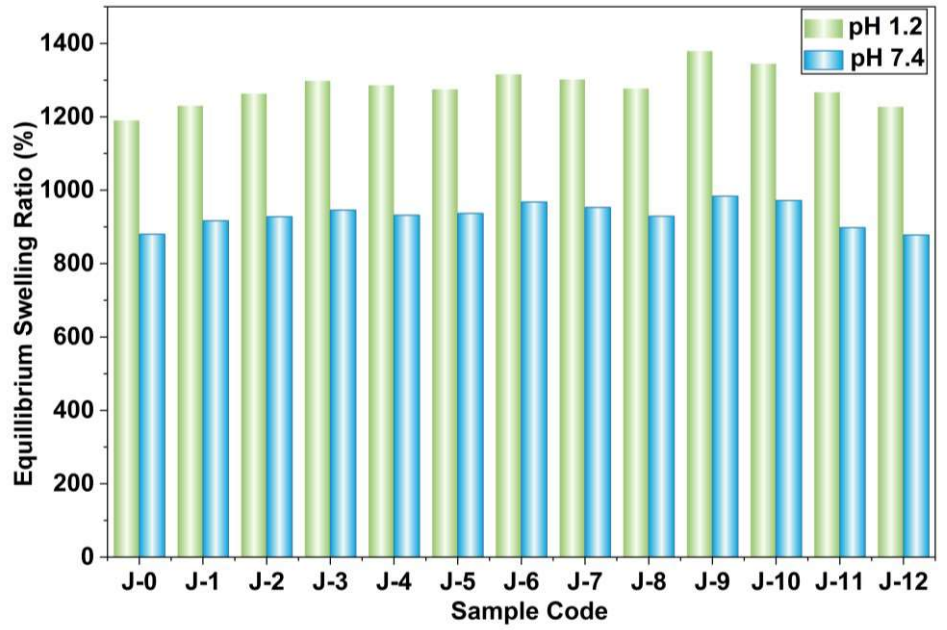

Figure 7. Swelling Ratio analysis of formulated hydrogel composites

4.4 Sol-gel content

The special water holding materials we made had a lot of gel and a little bit of liquid.Among these materials the one called hydrogel J-9 was the best at turning into a gel.This makes sense because hydrogel J-9 was also the best at soaking up water.This result shows that hydrogel J-9 has a strong network inside it.This strong network is probably because of the forces between the tiny parts of the material.These forces happen because of the groups called -COOH and -OH in the material.These groups are able to form bonds with each other like hydrogen bonds and other kinds of interactions.All these bonds together make the material

very stable and strong like a three- web.This is what makes hydrogel J-9 so good, at holding its shape and not falling apart.[37]

4.5 Drug Loading and Release Study

4.5.1 Efficiency of Drug Loading

The CMTKG/TG/PAM hydrogel, also known as J-9 was tested to see how well it could hold and keep drug molecules inside. The test showed that it was able to keep 65 percent of the drug molecules and hold 18 percent of the drug. This is a good result and it seems to happen because of the way the hydrogel and the drug molecules interact with each other. The hydrogel has a kind of attraction that helps keep the drug molecules trapped inside the hydrogel.

The CMTKG/TG/PAM hydrogel or J-9 seems like a choice for taking drugs by mouth. The people who made this test think that they can make the hydrogel even better at holding the drug molecules if they change how the hydrogel is made or if they add drug molecules to the hydrogel when they are making it. The CMTKG/TG/PAM hydrogel or J-9 is a candidate, for oral drug delivery because of the way it works with the drug molecules.

4.5.2 Release of Drug

The release of ampicillin from CMTKG/TG/PAM HG as shown in Figure 8 was much faster at pH 7.4 than at pH 1.2. This difference happens because of how the carboxylate groups behave at pH levels.At pH 7.4 these groups stay uncharged which makes the polymer chains move apart.This movement helps ampicillin come out of the hydrogel easily.At 1.2 the carboxylic acid groups get charged and form strong bonds with each other.

These bonds make the polymer chains come together tightly.This tight structure makes it hard for ampicillin to get out. It comes out slowly.The HG released ampicillin slowly and steadily over time.They kept ampicillin at levels in the body for a long time, which means people might not need to take it as often.The HG released 66% of ampicillin at pH 7.4 and 43% at pH 1.2.These results show that CMTKG/TG/PAM HG can be used to control the release of ampicillin in a way that responds to levels.The HG provide a way to deliver ampicillin in a controlled manner.CMTKG/TG/PAM HG can deliver ampicillin well.Ampicillin release

from HG happens at rates.The release of ampicillin, from CMTKG/TG/PAM HG is pH-dependent.

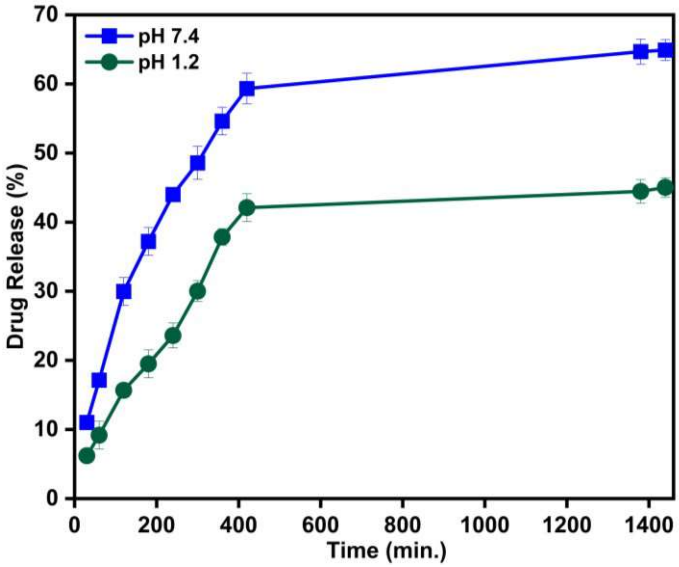

Figure 8. Cumulative drug release (%) versus time for ampicillin-loaded HG in media of pH 7.4 and pH 1.2

4.6 Drug Release Kinetics

Kinetics modeling was done on ampicillin-loaded CMTKG/TG/PAM hydrogel using release models. As shown in Figure 9 the release data matched best with the Korsmeyer-Peppas model in alkaline conditions. The Korsmeyer-Peppas model worked well for ampicillin release. At pH 7.4 the data fit well with $R^2 = 0.990$ and n = 1.275.This R^2 value shows a fit.The n value indicated Super Case II transport for ampicillin release.This meant that ampicillin release was mainly driven by polymer chain relaxation and erosion of CMTKG/TG/PAM hydrogel.In contrast at pH 1.2 the data fit well with $R^2 = 0.997$ and n = 0.73.The mechanism changed to Fickian diffusion for ampicillin release. Here, both diffusion and polymer relaxation-controlled drug release of ampicillin, from CMTKG/TG/PAM hydrogel.

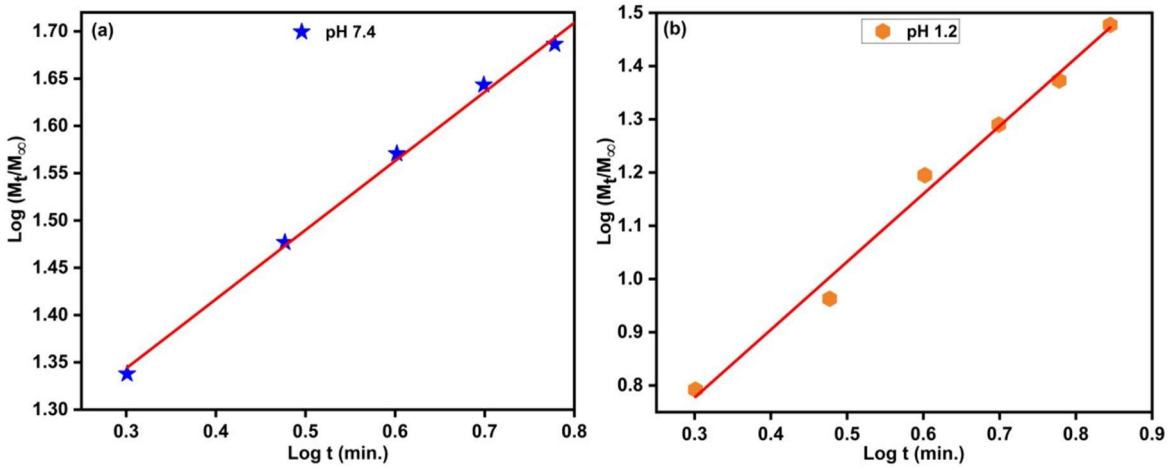

Figure 9. Linear regression plots of drug release data fitted to the Korsmeyer-Peppas model for the TG/PAM system under (a) pH 7.4 and (b) 1.2

CHAPTER 5

CONCLUSION

The CMTKG/TG/PAM hydrogel was made using a way called free-radical polymerization. This process used potassium persulfate to start the reaction and N N'- methylene bis-acrylamide as the thing that connects the parts together.The special hydrogel that responds to the level of acidity was then looked at closely to see if it could carry the medicine ampicillin.When the people making it tried things to make it work better they found out that if they used too much of the connector the hydrogel did not swell as much as it should.This happened because the parts were connected tightly so the hydrogel could not get bigger.The best hydrogel was very good with 92% of it being strong and not fallingOne of the mixtures called J-9 was the best at responding to the level of acidity and letting the medicine out.

At a level of acidity of 7.4, 66% of the medicine was released, which is what you want for delivering medicine to the intestines.The way the medicine was released was like the Korsmeyer-Peppas model, which's a way of describing how things move.At a level of acidity of 1.2 the medicine moved in a way and at 7.4 it moved in a very fast way.So the CMTKG/TG/PAM hydrogel is very good for giving ampicillin in a way that responds to the level of acidity. It could be very helpful for people who need this medicine. The CMTKG/TG/PAM hydrogel is a platform, for the pH-responsive delivery of ampicillin.