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Superabsorbent Polymer Gels based on Polyaspartic Acid and Polyacrylic Acid

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Abstract

Polymer gels based on polyaspartic acid (PAsp) and polyacrylic acid (PAA) have been synthesised using ethylene glycol dimethylacrylate (diacrylate-EGDMA) and Trimethylolpropane triacrylate (Triacrylate-TMPTA) as cross-linkers. Swelling behaviour of these polymers has been studied in different solutions like glucose, saline and water. The swelling behaviour of these polymers has also been studied under different pH conditions. The swelling capacity has also been analysed under load to have an idea of the gel strength (Absorbency under Load-AUL). Best absorbing characteristics, as indicated by the swelling behaviour, have been observed in case of polymer gels synthesized with EGDMA. Polymers with maximum PAsp have shown maximum superabsorbent properties in case of EGDMA as a cross-linker. However, with TMPTA as a cross-linker molar mass ratio of 1:2 polyaspartic acid: acrylic acid have shown better results. These polymers have better superabsorbent characteristics. TMPTA based polymers have shown better properties under load than EGDMA These polymers can be used as smart polymers for various applications e.g., drug delivery, materials for wound dressings, etc as they have shown varying behaviour in different conditions. The structure of the polymers has been studied by FTIR (Fourier Transform Infrared spectroscopy) and NMR (Nuclear Magnetic Resonance Spectroscopy). The surface morphology has further supported the results.

Keywords: Superabsorbent; Polymer gels; Swelling; Polyaspartic acid; Polyacrylic acid; Ethylene glycol dimethacrylate; Trimethylolpropane triacrylate

Introduction

Different decade gave different absorbing materials, each having their own specific absorption capacity. The commonly used water absorbing materials were based on cellulose based product like Whatman filter paper, tissue paper, wood pulp fluff, cotton ball with water absorption capacity (wt%) of 180, 400, 1200, and 1890, respectively. The water absorption capacity was 20 times their weight. The prominent disadvantage among these traditional absorbents was that they lose most of the fluids absorbed by them when they were squeezed. Due to this, a new class of speciality absorbents came into existence known as superabsorbent polymers (SAP). These synthetic polymers can be engineered to exhibit significant swelling when placed in water [1].

Superabsorbents developed are ultra-high absorbing material and could hold as high as 100% - 10000% (10 g/g - 1000 g/g) of water whereas the capacity of the hydrogels was 100% (g/g) [1]. They are a class of cross linked polymer capable of absorbing and retaining water. The cross linking present in SAP, does not allow SAP to dissolve but forms a gel when placed in water. The absorbency and swelling capacity are controlled by the type and degree of cross linkers used. Therefore, optimum cross linkers are used to develop superabsorbent polymers so that they exhibit high absorption capacity [1,2]. Due to their higher absorption and gel strength can lead to many fields like wound dressings, agriculture, wound care management, environment industry, diaper industry, etc [3-6].

PAsp has been reported to be used as polyelectrolyte and as a substitute for polyacrylic acid due to its biodegradability [7]. PAsp has been gaining importance in recent years as a biodegradable superabsorbent polymer [8-10]. PAsp based have found application as superabsorbent polymers in different industries mentioned above. Cross-linked PAsp, however has certain defects of poor strength due to high charge density along the polymer chains [11,12]. To improve upon the defects interpenetrating and semi-interpenetrating polymers

have been taken up by various scientists. A review has also been published on the polyaspartic acid based superabsorbents which cover the different types of polymers which include co-polymers, grafted and also interpenetrating polymers and semi-interpenetrating polymers [13]. Interpenetrating polymers involves networking of two different polymers as studied in case of poly (N-acrylamide) and polyaspartic acid to achieve functional materials which are pH sensitive and also thermo-sensitive [14,15]. Semi-interpenetrating polymers based on polyaspartic acid have been developed for drug delivery and other applications [16-19]. Some work on semi-interpenetrating network was initiated by Zhao et al. [20] in case polyaspartic acid and polyacrylic acid to achieve responsive polymers. With the upcoming newer areas, wherein these superabsorbent materials find application for biomedical applications like wound dressings, it has always been challenging to develop polymers with an ideal combination of water absorbency and gel strength. Thus, in continuation to the work reported by Zhao [20] different polymers based on PAsp and PAA have been synthesized using EGDMA, a difunctional cross-linker and TMPTA, a trifunctional cross-linker. The different polymers synthesized have been evaluated for their swelling properties.

Experimental

Materials

L-Aspartic acid and sodium hydroxide pellets (NaOH) were obtained from Merck Specialities Private Limited (Mumbai, India). Acrylic acid

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was purchased from Sigma Aldrich Chemical Corporation (St. Louis, USA). O-phosphoric acid for preparing the PSAP was purchased from Spectrochem Private Limited (Mumbai). Cross-linkers, Ethylene Glycol Dimethacrylate (EGDMA) and 1,1,1-Trimethylpropane triacrylate (TMPTA) were procured from Polysciences Inc. (Warrington, PA). Initiator ammonium peroxodisulphate (NH₄)₂S₂O₈ was purchased from Spectrochem Private Limited (Mumbai).

Preparation of polysuccinimide (PSI) and PAsp

L-Aspartic acid powder (5 g) and 85% of o-phosphoric acid (2.18 ml) were taken in a round bottom flask. The reaction mixture was heated for 3 h at 200°C under vacuum using rotary evaporator (Buchi type). Yellow powder of Polysuccinimide (PSI) was obtained which was then hydrolysed by adding sodium hydroxide (NaOH) solution prepared by dissolving 3.75 g NaOH in 25 ml deionised water. The NaOH solution was gradually added with continuous stirring by using magnetic stirrer to the PSI on an ice bath. To this mixture 35% HCl solution is added drop wise till it becomes neutral. This neutral solution was precipitated by adding saturated methanol solution (methanol in NaCl) and then filtered using vacuum filtration. The precipitate obtained is polyaspartic acid (PAsp) [20].

Synthesis of polymers containing polyaspartic acid (PAsp) and polyacrylic acid (PAA)

Preparation of neutral acrylic acid: Acrylic acid was neutralized by adding with NaOH solution (26 g NaOH in 100 ml of deionised water) drop wise using a dropping funnel. During addition of NaOH, the mixture was stirred on a magnetic stirrer to make a homogeneous mixture. The concentration of sodium hydroxide used results in 70% neutralization of the acrylic acid [21].

Preparation of polymers: Different volumes of aqueous polyaspartic acid (PAsp) and neutralised acrylic acid (Table 1) were taken and mixed for 10-15 min. In this study different ratios of acrylic acid and PAsp were used keeping the cross-linker and initiator concentration constant. The table shows the decreasing mass ratio of acrylic acid with relative increase in polyaspartic acid and with 40% water in all the systems studied. To these cross-linkers EGDMA and TMPTA were added in moles, followed by initiator ammonium peroxodisulphate. The mixture was heated to 80°C-100°C for 30 minutes under continuous stirring. Gel like product separated which was collected. The product obtained was analyzed for their swelling behaviour. The initiator concentration and cross-linker was established to give the best absorbent properties. The chemical structures of the cross-linkers used are given below in Figure 1. The reaction for the preparation of polymers with EGDMA and TMPTA is given below in Figure 2.

Analysis

Measurement of Molecular weight of Polyaspartic acid: Molecular weight of PASP was determined by using GPC and it was found to be 2,15,692. A homopolymer with polydispersity index Mw/Mn equal to 3.12 has been used for synthesizing the polymers.

Swelling in various physiological fluids: The swelling ratio of

SET.	PASP (%)	Acrylic acid (AA) (%)	Mass Ratio of PASP: AA	Initiator (g)	Cross- Linker (Moles)
1	10	50	1:5	0.009	0.05
2	20	40	1:2	0.009	0.05
3	30	30	1:1	0.009	0.05

Table 1: Different mass ratios of polyaspartic acid and acrylic acid studied.



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the various polymers was studied in various physiological fluids. The different physiological fluids used were water, saline solution and glucose solution. The main aim of using different fluids was to develop application of these polymers in biomedical area like wound dressings.

The different physiological solutions used for the study were:

- a) Saline Solution: Sodium chloride solution were prepared by dissolving 9 g NaCl/1000 ml
- b) Water: Conductivity of water was measured by conductivity meter. The conductivity of the water was 0.023μ S/cm and pH of water was neutral.
- c) Glucose solution-50 g glucose was dissolved in 100 ml deionised water.

The tea bags used in the method were made using mesh made of nonwoven polypropylene with dimensions of 15 cm by 15 cm. The SAP sample was dried and weighed. Around 0.15 g of sample was taken and weighed accurately. The sample of known weight was then placed in tea bag and this tea bag was weighed then immersed in different solutions. Three readings of each sample were taken. An average of three readings has been reported. The swelling ratio was calculated as:

Swelling ratio
$$\left(\frac{Wt}{Wt}\right) = Wt - W0 - Wn / W0$$
 (1)

Where, Wt is the weight of tea bag in grams including swollen polymer; W0 is the weight of dry sample in grams (g) and Wn is the weight of wet polypropylene mesh in grams (g). The swelling ratio is calculated at different intervals of time (0.5 h, 1 h, 2 h, 3 h, 4 h and 24 h).

Swelling in different pH solutions: Different buffer solution with various pH values (pH = 2, 4, 7, 8 and 10) were made by using NaOH and HCl. The pH values were checked by a pH meter by Decibel (accuracy = \pm 1). These were used to study the pH sensitivity of various polymers samples formed by using different cross linkers. The pH-sensitive properties of the polymer gels were studied in terms of swelling ratio by using tea bag method as discussed above. The same tea bag method was repeated for calculating swelling ratio of various polymers at different interval of time (0.5 h, 1 h, 2 h, 3 h, 4 h and 24 h).

Swelling/Absorbency under load measurement: A macro porous sintered glass filter plate (d = 100 mm, h = 7 mm) was placed in a Petri dish and the dry hydrogel sample was uniformly placed on the surface of polyester gauze located on the sintered glass. A cylindrical solid weight (d = 80 mm, variable height) which could slip freely in a glass cylinder was used to apply the desired load (applied pressure = 0.3 psi) to the dry hydrogel particles as shown in Figure 3. The sample was then covered by 0.5% saline solution such that the liquid level was equal to the height of the sintered glass filter.

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The dish and its contents were covered to prevent surface evaporation and probable change in the saline concentration. The swollen particles were weighed at regular time intervals and AUL was calculated by using equation (1):

AUL
$$(g/g) = W_2 - W_1/W_1$$
 (2)

Where, W_1 and W_2 represent the weight of dry and swollen hydrogel, respectively.

FTIR analysis: Perkin Elmer, FT-IR spectrophotometer-spectrum RX1 at from University Scientific Instrumentation Centre at University of Delhi was used to study FT-IR. The dried samples were ground into fine powder and then mixed with Potassium Bromide (KBr) powder. The mixed samples were compressed into pellets. The scanning wave number ranged from 4000 cm⁻¹ to 550 cm⁻¹.

NMR analysis: The NMR spectra of the various polymer gel samples were carried out in D_2O . 300 MHz NMR Spectrometer (Brucker NMR Spectrometer at Indian Institute of Technology, Delhi) was used for the study.

SEM analysis: SEM analysis was carried out on gold coated samples of the polymer gels. Analysis of gold coated samples was carried out using MODEL Carl Zeiss SEM Analyser, EVO-18. The magnification used for the study was 250 X.

Results and Discussions

Neutralised acrylic acid is used to prepare the SAP samples with polyaspartic acid. Acrylic acid monomer is usually neutralised by alkali (NaOH) owing to high activity of acrylic acid over sodium acrylate. This neutralisation degree of acrylic acid has been reported to affect the swelling capacity of SAP and at an optimum value of degree of neutralisation is 65%-70% the swelling capacity as reported [21]. The

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best swelling is observed due to maximum repulsion between the – COO^{-} and $-COO^{-}Na^{+}$ groups, thus resulting in breaking of cross-links. Above 65%-70% degree of neutralization the swelling decreases due to further increase in electrostatic repulsion between $-COO^{-}$ and $-COO^{-}Na^{+}$. Due to increase in repulsion, the cross -links in the polymer further decreases thereby decreasing the water entrapping capability. Similarly, when degree of neutralisation is lower, the cross-linking is higher and swelling is lower. Thus an optimum degree of neutralization is required to obtain good swelling.

Swelling in various physiological fluids

The observation and results of the swelling in different physiological fluids are discussed below.

Water: The results of the study are shown in Figure 4. The results have shown that with increasing time the swelling is better in case of EGDMA as a cross-linker than TMPTA. In case of TMPTA the swelling characteristics have shown an increased initial absorption being higher in all ratios studied. But in case of EGDMA as a cross linker difference in swelling characteristics have been observed. The initial absorption is low in comparison to TMPTA but a continuous increase in absorption has been observed with time. The 24 h swelling is better in case of EGDMA than TMPTA for all ratios studied. The 24 h absorption in case EGDMA is ~1100 g/g, while in case of TMPTA it has been observed to be ~200 g/g for polymers with maximum polyaspartic acid. Even in case of polymers with maximum acrylic acid (1:5) where EGDMA has shown best results the swelling ratio is 800 g/g while in case TMPTA it is 300 g/g. Thus, the increasing concentration of polyaspartic acid has resulted in better absorption in case of EGDMA and a reverse trend has been observed in case of TMPTA i.e in case of TMPTA polymers with minimum concentration of Polyaspartic acid and maximum concentration of acrylic acid have shown better swelling characteristics.

Saline solution: The swelling characteristics of the polymers in saline solution have shown different results. Figure 5 shows the trends observed in case of saline absorption. The overall swelling of polymers in saline is lower than that observed in water. The polymer samples with EGDMA as a cross-linker and high concentration of polyaspartic acid have shown increased swelling after exposure for 24 h. But for TMPTA as cross linker totally reverse trend has been observed. The initial swelling of polymer samples with EGDMA is better with increased concentration of polyaspartic acid. In case of TMPTA however the initial absorption of saline is better in case of maximum acrylic acid. The polymers with maximum polyaspartic acid has shown a maximum absorption of ~ 82 g/g for 24 h absorption in case of EGDMA while in case of TMPTA it is 40 g/g. Polymers with maximum acrylic acid in case of EGDMA and TMPTA have shown same swelling ratio of ~ 70 g/g for 24 h absorption. The swelling ratio in case EGDMA and TMPTA with 1: 2 ratio is ~ 32 g/g and 42 g/g, respectively.

The trend observed in case EGDMA and TMPTA are different. The initial swelling, upto 3 h, is better for polymers with increased polyaspartic acid than with acrylic acid with both the cross-linkers. After 3 h the trend in swelling ratios changes and the swelling increases in case of maximum polyaspartic acid in case of EGDMA based polymers.

Glucose solution: The Figure 6 explains that the swelling of polymers in glucose increases with time for all the concentration of Polyaspartic acid and acrylic acid studied with both crosslinkers. The swelling capacity has been observed to increase as the concentration of polyaspartic acid increases for both cross linkers. Best result have been observed with EGDMA cross-linked polymers with low concentration







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of acrylic acid & high concentration of polyaspartic acid for 24 h absorption. In case of TMPTA however best results have been observed in case if 1: 2 polyaspartic acid to acrylic acid ratio studied. Minimum swelling has been observed in case of polymers with maximum polyaspartic acid. The maximum swelling ratio observed was 175 g/g in case of TMPTA for 24 h and 550 g/g in case of EGDMA. Thus, the swelling in different physiological fluids can be concluded that EGDMA has given better absorption for all fluids like deionised water, glucose & saline solution.

Swelling at different pH: Figures 7 and 8 shows the effect of changing pH on the swelling characteristics of polymers synthesized using EGDMA and TMPTA as cross-linkers, respectively. In case of pH 2, the polymer with EGDMA as cross-linker has given better results than TMPTA. At acidic pH both EGDMA and TMPTA with high acrylic acid ratio i.e. 1: 5 has shown better swelling.

At pH 4, the EGDMA based polymers has shown better swelling than TMPTA. EGDMA based polymers with the ratio1: 2 and 1: 5 polyaspartic acid: acrylic acid has shown almost equal swelling after 24 h whereas EGDMA with a ratio of 1:1 polyaspartic acid: acrylic acid has shown different results. However, in case of TMPA polymers with maximium acrylic acid ratio has shown maximum swelling followed by polymers with ratio of 1: 2 and 1: 1 (polyaspartic acid: acrylic acid).

At pH 7, the polymer with EGDMA as crosslinker has shown better swelling than TMPTA. The polymer with EGDMA as cross-linker and ratio of 1: 1 shows highest swelling ratio. However in case of TMPTA similar results have not been observed with the similar ratio of 1: 1. At pH 8, EGDMA and TMPTA with 1: 1 ratio of polyaspartic acid: acrylic acid has shown higher swelling than rest of the ratios studied. At this pH, polymers with both the crosslinker have shown increased swelling capacity due to the presence of polyaspartic acid.

At basic pH, i.e.10, polymers with TMPTA as crosslinker interact and swell well for all ratios studied than EGDMA. The later behaves poorly and gives very low swelling. Both EGDMA and TMPTA shows varied absorption pattern at different pH. But EGDMA as a cross-linker has proved to result in polymers with better superabsorbent polymers than TMPTA. This study shows that for application under alkaline conditions, polyaspartic acid polymer with acrylic acid and EGDMA should be a better choice than TMPTA. One such area where it can find application is in highly exudating wounds. Both TMPTA and EDMA based SAPs possess same functional group. The EGDMA and TMPTA are having COO⁻ (EGDMA, TMPTA and acrylic acid). When both types of SAP were exposed to acidic and alkaline condition, swelling was observed.

The functional groups present in these polymers are -COOH, -COO'Na+, -CONH- peptide bonds and -COOC- ester bonds. These groups are affected by the pH conditions. Under acidic conditions the peptide bond gets protonated forming -CONH₂⁺ causing repulsion between -COO⁻Na⁺ and -CONH_a⁺ thus affecting the swelling properties. The ester bond present in the cross-linkers results in hydrolysis thus decreases the cross-links and probably results in enhancing the swelling. However under alkaline conditions the peptide bond and the ester bond hydrolyse giving better swelling. An optimum level of absorbency has been achieved in case of both cross-linkers from pH 7-8. At pH 10 however the water holding capacity is decreased due to decrease in cross-links. The greater the quantity of Polyaspartic acid the more -CONH₂⁺ thus increased repulsion and therefore better swelling. However in case of trifunctional cross-linker the cross-linkage formed are more than the with the difunctional cross-links thus responsible



Figure 7: Effect of pH on the swelling ratios of polymers with EGDMA as cross-linker



for different trends observed. These are further observed with swelling characteristics observed under load.

Under saline conditions the -COONa groups are formed that which decreases the swelling and does npt allow flow of saline into the polymers thus a decrease in swelling is observed. In case of glucose, the swelling is better than saline and less than water because in glucose solution the water is associated with glucose through hydrogen bonds. Thus the movement of glucose into the polymer is relatively slow in comparison to water thus decrease in swelling has been observed.

Swelling/Absorbency under load: The results of absorbency under load are given in Table 2 below. The absorbency under load observed for the polymers is different from the results otherwise. The absorbency under load for saline solution is lower than that observed under normal conditions. It can further be observed that TMPTA based polymer samples has shown better results than EGDMA based polymers. It can further be indicated that the gel strength is better in case of TMPTA than that observed in case of EGDMA. The best gel strength from the above results in case of TMPTA with 1: 2 ratio. Therefore to obtain polymers with good strength and absorbency TMPTA could be chosen with ratio of 1: 2 polyaspartic acid and arcylic acid. In case EGDMA

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S.No	Sample	AUL (g / g)	Sample	AUL (g / g)
1.	EGDMA (1:5)	13.98	TMPTA(1:5)	18.57
2.	EGDMA (1:2)	13.00	TMPTA(1:2)	24.62
3.	EGDMA (1:1)	13.02	TMPTA(1:1	18.09

Table 2: Results of absorbency under load for the polymer gel samples.

although comparative result has been observed with 1:2 and 1: 1 but absorbency is better with 1: 1. Thus depending upon the absorbency desired the cross-linkers can be selected. This is further supported by the results of grafting which has been reported to be higher in case of TMPTA i.e.102% while in case of EGDMA is 65%, when extracted with chloroform for 24 h in a soxhlet apparatus in case of samples with 1:1 ratio of polyaspartic acid and acrylic acid

FTIR spectroscopy: The IR studies have been presented for the polymer with all concentration of polyaspartic acid and acrylic acid. The structure and bonding of the polymer has been proved by this study. Figures 9 and 10 gives the IR of the sample prepared with EGDMA, TMPTA as a cross-linker, respectively. The FTIR studies are representing the bonding between the polymeric chains and the various functional groups present. The IR of the sample further shows similar types of peak of the carboxylate and carboxylic. The presence of -CH₂ group in polyaspartic acid, EGDMA, TMPTA and neutralised acrylic acid is shown at 2923 cm⁻¹-2943 cm⁻¹. Bending vibration of -CH, group has been observed at 1402 cm⁻¹- 1462 cm⁻¹. The -NH, group is confirmed by > NH₂ stretching at 2343 cm⁻¹- 2375 cm⁻¹ which is due to the presence of polyaspartic acid. It has also shown peak for C-O-C bond as observed in case of both EGDMA at 1070 cm⁻¹- 1085 cm⁻¹ and TMPTA at 1065 cm⁻¹-1090 cm⁻¹. The presence of -C=O (carbonyl group) at 1717 cm⁻¹729 cm⁻¹ and C-O-C bonding has been observed formed due to ester groups.

The NMR spectra for the different polymers are presented in Figure 11 for polymers with EGDMA and Figure 12 for polymers with TMPTA, respectively. The peak values at 1.5 ppm and 1.4 ppm is for alkyl groups i.e., R_3 CH- and R_2 CH- respectively. The presence of polyaspartic acid is confirmed by the peak at 2.1 ppm (2H,t) and 2.3 ppm (2H,t). The cross linker and polyaspartic acid are linked by an ester bond shows a peak at 3.6 ppm for EGDMA based polymers but for TMPTA this linkage is prominent for ratio 1: 1 and 1: 2. It might be because cross linker TMPTA is bonding well and easily with acrylic than polyaspartic acid. Due to this TMPTA (1: 5) has high swelling among all three ratios. The peak at 2.2 ppm corresponds to -CH₂. present in EGDMA (-C(=0)-CH₂-CH₂-C(=O)-).

SEM analysis: The SEM studies have been carried for all concentration of Polyaspartic acid and acrylic acid with both crosslinkers EGDMA and TMPTA. Figure 13 presents the micrograph of polymers with TMPTA as cross linker and Figure 14 gives the result of EGDMA based polymers. The scanning electron micrographs have shown that the polymers obtained with these cross-linkers are different. Figure 13 shows that the porosity in case of polymers with 1: 5 and 1: 1 ratio of polyaspartic acid: acrylic acid in case of TMPTA while in case of samples with ratio of 1: 2 the samples have relatively low porosity which also evident from the results of swelling as well from the AUL data. Figure 14 shows that in case of EGDMA samples with ratio of 1: 1 the samples have shown porosity as compared to the other two ratios studied. Moreover the pores observed are macropores in EGDMA that those observed in case of TMPTA. Due to these pores only the swelling has increased to approximately 1000 g/g in distilled water. As the concentration of polyaspartic acid increases pores are formed on the surface which allow seepage of fluids like water, saline solution, glucose



Figure 9: IR spectra of polymer samples with EGDMA as cross-linker.







solution which into the polymer forming superabsorbent polymer (SAP).

Conclusions

The superabsorbent polymers prepared by replacing acrylic





Figure 13: The scanning electron micrographs of samples with TMPTA as a cross-linker at 250X; (a) TMPTA 1 with ratio 1:5 of PAsp and PAA; (b) TMPTA 2 with ratio 1:2 of PAsp and PAA and (c) TMPTA 3 with ratio 1:1 of PAsp and PAA.



Figure 14: The scanning electron micrographs of samples with EGDMA as a cross-linker at 250X; (a) EGDMA 1 with ratio 1:5 of PAsp and PAA; (b) EGDMA 2 with ratio 1:2 of PAsp and PAA and (c) EGDMA 3 with ratio 1:1 of PAsp and PAA.

acid with polyaspartic acid using different cross linkers have shown maximum swelling capacity in deionised water and minimum in case of saline solution for 24 h absorption studies. The trend observed in different physiological fluids is as follows: Distilled water > glucose solution > saline solution (EGDMA) and glucose solution > distilled water > saline solution (TMPTA).

The polymer properties can thus be tailored and used for different applications. EGDMA based polymers have shown better absorption properties than TMPTA. But the results of AUL has shown that TMPTA has much better strength than EGDMA based sample especially the polymer with 1: 2 ratio of polyaspartic aid and acrylic acid.

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