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Synthesis and Characterization of Malonic Acid Crosslinked Chitosan-Sodium Carboxymethyl Cellulose Mixed Hydrogel and Studies on Its Probable Use as Micro Water Reservoir in Soil

M. DAS[1,*], S. BARKATAKY[2], D.K. KAKATI[1] and B. GOGOI[2,*]

[1]Department of Chemistry, Gauhati University, Guwahati-781014, India
[2]Department of Geology, Gauhati University, Guwahati-781014, India

*Corresponding authors: E-mail: mitudas2603@gmail.com; bikash.gogoi@gauhati.ac.in

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Climate change has impacted the worldwide rainfall pattern resulting in either too little or too much rainfall. This has adversely affected the cultivation of water intensive crops like rice. This calls for judicious irrigation process avoiding wastage of water. Use of hydrogels in soil acting as *in situ* water reservoirs, releasing water to soil in a slow manner may address the problem of wastage of water and associated leaching of nutrients from soil. The present work describes the synthesis of a mixed hydrogel from two polysaccharides chitosan (chit) and sodium carboxy methyl cellulose (NaCMC) in presence of malonic acid (MA) as crosslinking agent. The hydrogel samples were characterized using FTIR, TGA, PXRD and SEM techniques. The swelling studies indicated approximately 1700% swelling at pH 9.2. Swelling index values for the hydrogel was found to increase from acidic to alkaline medium. The water holding capacity was investigated using soil samples collected from five different locations including Kamrup (Metro), Kamrup (Rural), Nagaon, Nalbari and Bajali districts of Assam, India which were classified as sandy soil. The experimental results revealed high water holding capacity for the soil samples in the pH range of 6.0-7.0. Experimental evaluation on water holding also indicated that hydrogel addition to the soil samples significantly increased its water holding capacity compared to the untreated soil samples.

Keywords: Judicious irrigation, Mixed hydrogel, Swelling index, Water holding capacity.

INTRODUCTION

Insufficient and haphazard rainfall affects the availability of water for a healthy agricultural production. So, it is essential to avoid wastage and sensible use of available water to gain maximum benefit. In other words, it is of utmost importance to prioritize the management of irrigation process. In this connection, a novel idea has immersed where biodegradable materials with capability of high water absorption, retention and slow release of absorbed water in time are being used in soil for more effective use of irrigation water [1,2]. Biodegradable hydrogels are the most promising materials, which when used in soil may effectively act as tiny water reservoirs, storing water when it is plenty and releasing when water quantity goes down [3].

Hydrogels are three dimensional crosslinked materials produced from hydrophilic polymers with both physical and chemical crosslinks. They can absorb a large amount of water without undergoing any structural damage. The hydrophilic polymers having groups such as -COOH, -OH, -$CONH_2$, -CONH and -SO_3H are capable of producing hydrogels under favourable reaction conditions [4]. Although hydrogels from synthetic polymers constitute the major portion of commercial hydro-els, there is a growing interest on hydrogels from biopolymers due to their low cost, biodegradability, biocompatibility and renewable nature [5]. The polysaccharide hydrogels based on cellulose, chitosan, starch, alginate, dextrin, *etc.* are creating keen interest due to their environment friendly nature and have shown wide ranging applications in agriculture, medicine, pharmaceuticals, biotechnology, *etc.* [6,7].

The most abundant biopolymer in nature is cellulose. Carboxymethyl cellulose, obtained by replacing the hydroxyl groups on glucopyranose chains of cellulose with the carboxymethyl group (-CH_2COOH), is the most abundantly used water soluble anionic cellulose derivative, which can be easily crosslinked and used for hydrogel preparation [8,9]. Due to its low cost, high abundance, biocompatibility, sensitivity to pH, good water solubility and ionic strength, carboxymethyl cellulose finds applications in preparation of hydrogels with superabsorbent properties. However, the major disadvantage of the

three-dimensional polymeric network of carboxymethyl cellulose is its weak mechanical strength resulting out of the intramolecular crosslinks within the polymeric network rather than intermolecular crosslinks. To overcome this limitation, carboxymethyl cellulose is blended with other polymers to stabilize the polymeric structure of the hydrogel [10,11]. Chitosan, derived from the deacetylation of chitin and mostly from the cell walls of fungi and crustaceans, is a cationic polysaccharide capable of establishing a strong linkage with oppositely charged materials, thereby forming a polyelectrolyte complex [12]. Chitosan is composed of glucosamine and N-acetyl glucosamine units connected by β-(1-4)→ linkage and are the most abundant natural polymer characterized by its nontoxic, biocompatible and biodegradable nature with antimicrobial activity, which is widely used in agricultural sector for the protection of crop owing to its effects on the plants response as an elicitor [13,14]. However, the swelling capability of chitosan-based hydrogels is comparatively lower due to the slower relaxation rate of the polymer chains which can be improved by blending chitosan with other hydrophilic polymers at the gel state [15].

Several works have been reported on the synthesis of chitosan/carboxymethyl cellulose blend hydrogels and its application in drug delivery [16,17], adsorption [18-21], response in electric field [22], thermosensitivity [23], agriculture [24], *etc.* In this work, we aim to synthesize an eco-friendly mixed hydrogel from **s**odium carboxymethyl cellulose and chitosan crosslinked with malonic acid as crosslinker and investigate the efficiency of the prepared hydrogel for possible use in sandy soil samples as micro water reservoir. Sandy soils, distributed globally plays a very significant role in agricultural sector, especially with urbanization and the expanding population [25]. But sandy soils are generally characterized by low water holding capacity. Therefore, sandy soils are best benefited by the presence of hydrogels which can imbibe a large volume of water to its original volume. The application of hydrogel can increase water retention capacity of soil by 50-70% with varying dosages of soil to hydrogel ratio [26]. Application of hydrogels cannot only enhance the water use efficiency and irrigation intervals, but also decreases the cost of irrigation and provides the plants with desired nutrients and moisture content as per demand. When the soil around plant root gets dried out, the absorbed amount of nutrients and water by the hydrogel gets slowly released into the soil making them available for the plants [27].

Swelling behaviour of the hydrogel will also be investigated in this work to elucidate the effect of pH on swelling and thereby three different pH media with pH 4.0, 7.0 and 9.2 will be considered. The prepared hydrogel sample will be analyzed using FTIR, PXRD, TGA and SEM techniques and the hydrogel sample will be designated as Chitcmc throughout the study.

EXPERIMENTAL

The polymers sodium carboxymethyl cellulose (CMCNa, Merck) and chitosan (Chit, medium MW, SRL) were used for the preparation of the hydrogel. Malonic acid (MA, Loba Chemie) was used as the crosslinker. All the experiments were carried out in double distilled water.

Characterization: The FTIR spectra of the hydrogel samples were recorded on BRUKER ALPHA II spectrophotometer with attenuated total reflectance (ATR) crystal sampler in the range 4000-500 cm^{-1} and all the data were taken in transmittance mode. The PXRD patterns were obtained using Powder X-Ray Diffractometer (Empyrean PANalytical) over the scanning range of 2θ = 5-80º with Cu$K\alpha$ (λ = 1.54 Å). The thermogravimetric analysis (TGA) of the hydrogel sample was done using thermogravimetric analyzer Mettler Toledo TGA-2 in the temperature range 25-800 ºC and at a heating rate of 10 ºC/min for each sample with nitrogen flow of 20 mL/min. The surface morphology of the hydrogel film was examined using a field emission scanning electron microscope (ZEISS SIGMA 300) at an accelerating voltage of 15 kV. The samples were coated with a thin layer of gold before analysis.

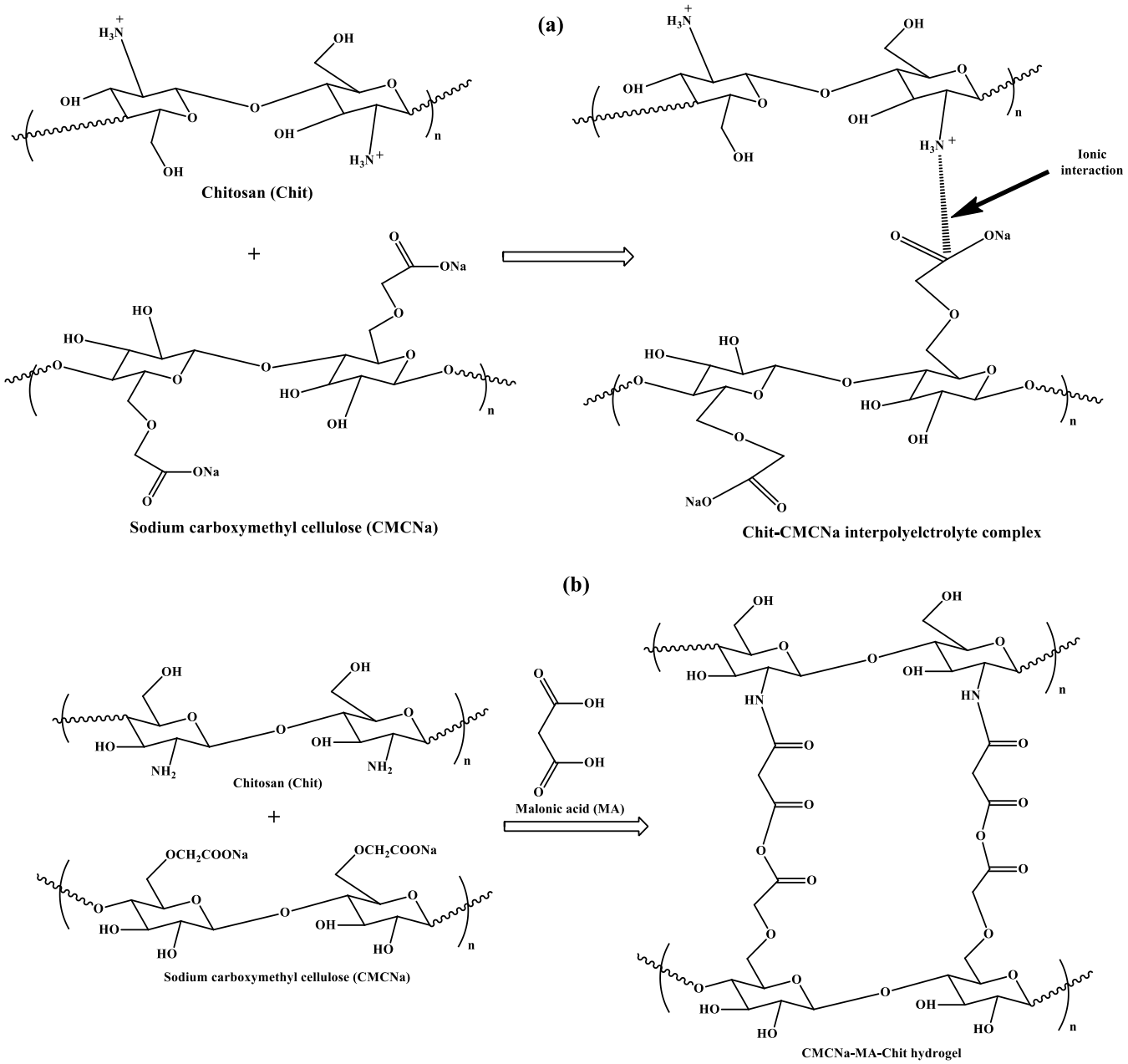
Synthesis of hydrogel: The procedure followed for preparation of the Chitcmc hydrogel is a partially modified process of Uyanga *et.al* [28]. A total polymer concentration of 2% (w/v) was maintained for the hydrogel preparation wherein the ratio of chitosan to sodium carboxymethyl cellulose was 1:3 and the concentration of crosslinker malonic acid taken was 20% (% w/w total polymer). Initially, chitosan was dissolved in 50 mL of 1% acetic acid solution under stirring at 300 rpm at 80 ºC. CMCNa and MA were dissolved in 50 mL distilled water under stirring at 300 rpm at room temperature. Finally, both the solutions were mixed and stirred for 1 h for the hydrogel formation. The resultant hydrogel was poured into petri dish and dried under vacuum at 40 ºC for 24 h to remove the entrapped gas molecules if any. The sample was further dried at 70 ºC for another 24 h in hot air oven. Finally, the as-prepared hydrogel film was washed with distilled water and then with isopropyl alcohol to remove the water entrapped followed by drying it at 40 ºC for 4 h in hot air oven. The films were stored in a vacuum desiccator for further use (**Scheme Ia-b**).

Swelling ratio studies: The swelling behaviour of the hydrogel sample was studied in buffer solutions of pH 4.0, 7.0 and 9.2. For this, a known amount of completely dried preweighed hydrogel sample was immersed in a certain known volume of the respective solution at room temperature. The sample was removed at predetermined time intervals, excess water adhered to the surface was wiped out using a tissue paper and weighed accurately in a WENSAR, HPB310 electronic balance followed by immersing it again in the respective swelling medium. The swelling ratio (%) of the hydrogel was determined from the following equation:

$$\text{Swelling ratio (\%)} = \frac{W_S - W_D}{W_D} \times 100 \quad (1)$$

where W_D denotes the weight of dry hydrogel and W_S denotes the weight of swollen hydrogel.

Determination of soil's pH: In order to determine the pH of the soil samples, initially slurry was prepared using 1:1 ratio of air-dried soil and distilled water. The samples were mechanically stirred for 1 h to get the corresponding slurry and then kept undisturbed to a period of 48 h. After 48 h, the soil-water suspension was swirled mildly and the pH of the soil was recorded using ECOPHTEST1 pH meter (Eco Testr



Scheme-I: (a) Formation of Chit-CMCNa interpolyelectrolyte complex (b) Cross-linking between CMCNa and Chit with MA as a cross linker

Ph 1 Tester). After each reading, the electrode was rinsed thoroughly with distilled water and the excess water was blot off before further use for the next sample [29].

Grain size analysis: The distribution of each grain size within the collected soil samples was investigated using the previously reported method of grain size analysis by Folk & Ward [30]. Briefly, the air-dried samples were initially disintegrated by applying figure pressure and 100 g of each soil sample were sieved in a column of sieve with mesh size ranging from 2.8-0.062 mm *i.e.* -1.5 φ to 4 φ as per Wentworth scale [31] with an interval of 0.5 φ, respectively. The mechanically separated sieved grains were collected and weighed accurately to determine the distribution of sand and some amount of coarse silt present in the soil samples. The material collected from the base pan of the column were also weighed and used for further pipetting. The process of pipetting was employed to determine the distribution of some amount of medium silt and the clay size materials present in the soil samples. The weighed materials were put in a 1 L graduated cylinder and following the time interval as well as procedures as proposed by Carver [32], a total of eight samples were collected by the pipette stem and the values so obtained have been tabulated in Table-1.

The collected samples were oven dried at 100 ºC and weight difference between the samples with beaker and the empty beaker was considered as the distribution of samples of grain size ranging from some amount of 4.5 φ to 10 φ *i.e.* some amount of medium silt and the clay size material. The measured weight frequency values of grain sizes were recalculated to 100% and the cumulative weight frequencies of the

TABLE-1
PIPETTE WITHDRAWAL TIME CALCULATED FROM STOKES LAW AFTER CARVER [Ref. 32]

Diameter in Phi scale	Withdrawal depth (cm)	Elapsed time for withdrawal of sample in hours (h), minutes (m) and seconds (s)									
		18 ºC	19 ºC	20 ºC	21 ºC	22 ºC	23 ºC	24 ºC	25 ºC	26 ºC	27 ºC
4.0	20	20s	20s	20s	20s	20s	20s	20s	20s	20s	20s
4.5	20	2m 0s	1m 57s	1m 54s	1m 51s	1m 49s	1m 46s	1m 44s	1m 41s	1m 39s	1m 37s
Restir											
5.0	10	2m 0s	1m 57s	1m 54s	1m 51s	1m 49s	1m 46s	1m 44s	1m 41s	1m 39s	1m 37s
5.5	10	4m 0s	3m 54s	3m 48s	3m 42s	3m 37s	3m 32s	3m 27s	3m 22s	3m 18s	3m 13s
6.0	10	8m 0s	7m 48s	7m 36s	7m 25s	7m 15s	7m 5s	6m 55s	6m 45s	6m 36s	6m 27s
7.0	10	31m 59s	31m 11s	30m 26s	29m 41s	28m 59s	28m 18s	27m 39s	27m 1s	26m 25s	25m 49s
8.0	5	63m 58s	62m 22s	60m 51s	59m 26s	57m 58s	56m 36s	55m 18s	54m 2s	52m 49s	51m 39s
9.0	5	4h 16m	4h 9m	4h 3m	3h 58m	3h 52m	3h 46m	3h 41m	3h 36m	3h 31m	3h 27m
10.0	5	17h 3m	16h 38m	16h 14m	15h 50m	15h 28m	15h 6m	14h 45m	14h 25m	14h 5m	13h 46m
11.0	5	68h 14m	66h 32m	64h 54m	63h 20m	61h 50m	60h 23m	58h 59m	57h 38m	56h 20m	55h 5m

grain's sizes were plotted in arithmetic probability graph following Folk & Ward's method [30]. The values of φ5, φ16, φ25, φ50, φ75, φ84 and φ95 were evaluated from the prepared graph and used for mathematical calculation of graphic mean grain size (M_z), graphic standard deviation (σ_I), graphical inclusive skewness (S_{ki}) and graphical kurtosis (K_G), respectively.

Water holding capacity (WHC): Keeping in view the effect of soil type and texture on water holding capacity, the soil samples collected from various place Kamrup (Metro), Kamrup (Rural), Nagaon, Nalbari and Bajali districts of Assam state, India were investigated for determining the water holding capacity of the Chitcmc hydrogel film in soil. In order to carry out the investigation, an experimental set up was arranged wherein four beakers were filled with 10 g of soil sample each. A known amount of pre-weighed hydrogel sample 0.1% (w/w), 0.2% (w/w) and 0.3% (w/w) were mixed with soil samples in three of the beakers while the fourth beaker without the hydrogel sample acted as blank. Four different glass tubes of diameter 2 cm, sealed with filter paper at one end, were taken and the respective soil samples were carefully pourend into the glass tubes and weighed. Thereafter water was slowly added from top of the glass tube until the filter paper at the bottom just gets wet. The glass tube was weighed again after the addition of water. The water holding capacity (%WHC) by the Chitcmc hydrogel mixed soil sample was then evaluated using the following equation:

$$\text{WHC}\ (\%) = \frac{W_2 - W1}{W_0} \times 100 \qquad (2)$$

where W_0 denotes weight of pure soil; W_1 denotes weight of soil sample mixed with hydrogel; W_2 denotes weight of soil sample mixed with hydrogel after addition of water.

RESULTS AND DISCUSSION

Chitosan is a cationic polymer and sodium carboxymethyl cellulose is an anionic polymer. The mixing of these oppositely charged polymers leads to the formation of polyion or interpolyelctrolyte complex [33]. However, on incorporation of malonic acid as a crosslinker into the polyelectrolyte matrix, it tends to form crosslinks with chitosan and sodium carboxymethylcellullose *via* the $–COO^-$ and –OH groups attached to the crosslinker. The crosslinking follows an esterification mechanism leading to the formation of a stabilized 3D crosslinked structure of the hydrogel. The formation of Chit-CMCNa interpolyelctrolyte complex and the crosslinking between CMCNa and Chit with MA as a crosslinker is represented by **Scheme Ia-b**, respectively [28,34].

FTIR studies: In the FTIR spectrum of CMCNa, the peaks at 3684, 3486, 3371, 3194 cm^{-1} can be attributed to the O–H stretching. Furthermore, the peaks observed at 2922, 1583, 1410, 1027 and 669 cm^{-1} can be assigned to the C–H stretching, asymmetric COO^- stretching, symmetric COO^- stretching, C–O–H stretching vibration of alcoholic group and C–O–H bending vibration, respectively [35,36]. The major peaks observed for Chit are at 3600, 3490, 3278 cm^{-1} (O–H *str*. overlapped with N–H *str*.), 2885 cm^{-1} (C–H *str*.), 1639 cm^{-1} (C=O *str*.), 1540 cm^{-1} (N–H *bend*.), 990 cm^{-1} (C–N *str*.), respectively [37,38] (Fig. 1a). In the FTIR spectrum of MA, the absorption bands observed at 3462, 3194 cm^{-1} can be attributed to O–H stretching vibration. The peaks observed at 1707, 1637, 1400 cm^{-1} are due to C=O stretching, hydrogen bonded C=O stretch, symmetric COO^- stretching and the peaks at 1293 and 1163 cm^{-1} can be attributed to C–O stretching, respectively [39-41] (Fig. 1b). The FTIR spectrum of the Chitcmc hydrogel film had most of the absorption peaks of the constituents, with little variation in certain absorption peaks like at 990 cm^{-1} for chit and 1583 and for CMCNa indicating interaction between $–COO^-$ and $–NH_3^+$ groups (Fig. 1c). Furthermore, the absorption peaks at 1410 cm^{-1} was observed to weaken for the crosslinked Chitcmc hydrogel owing to the reduction in the number of –COOH groups in the polymeric network of CMCNa indicating a successful crosslinking by MA crosslinker [42] (Fig. 1d).

PXRD studies: In the XRD pattern of CMCNa, a single intense peak was observed at $2\theta = 20^\circ$ indicating poor crystalline nature (Fig. 2a), whereas the diffraction pattern for Chit is attributed to its hydrated crystalline structure (Fig. 2b). Comparing the XRD pattern of Chitcmc hydrogel film with CMCNa and Chit, the intense peak observed at 20º and 19.9º for CMCNa and Chit was found to transform to a shoulder peak (Fig. 2c). This may be due to loss of even the poor crystalline nature of the components in the hydrogel for the

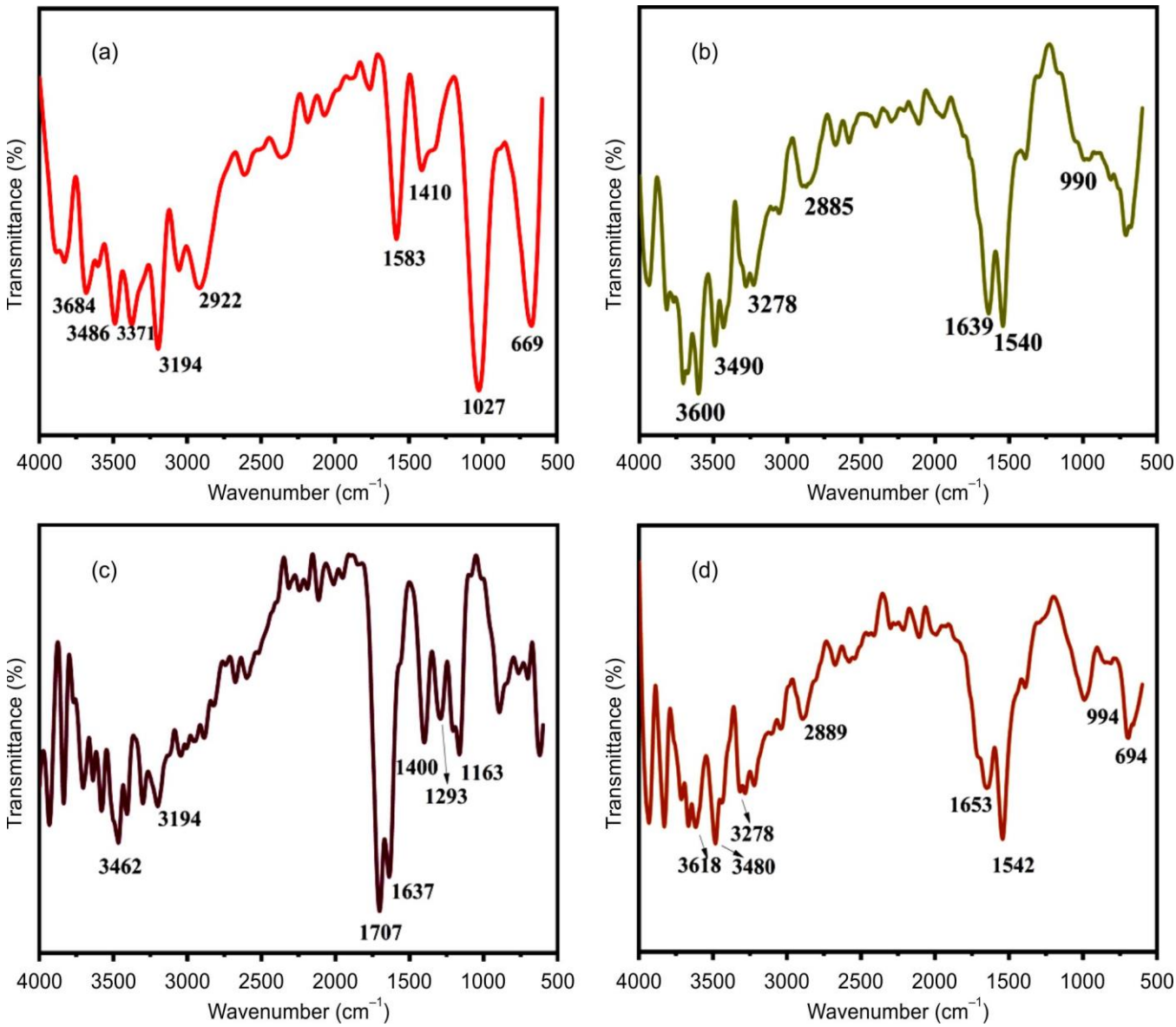


Fig. 1. FTIR spectra of (a) sodium carboxymethyl cellulose (CMCNa) (b) chitosan (Chit) (c) crosslinker malonic acid (MA) (d) crosslinked hydrogel film (Chitcmc)

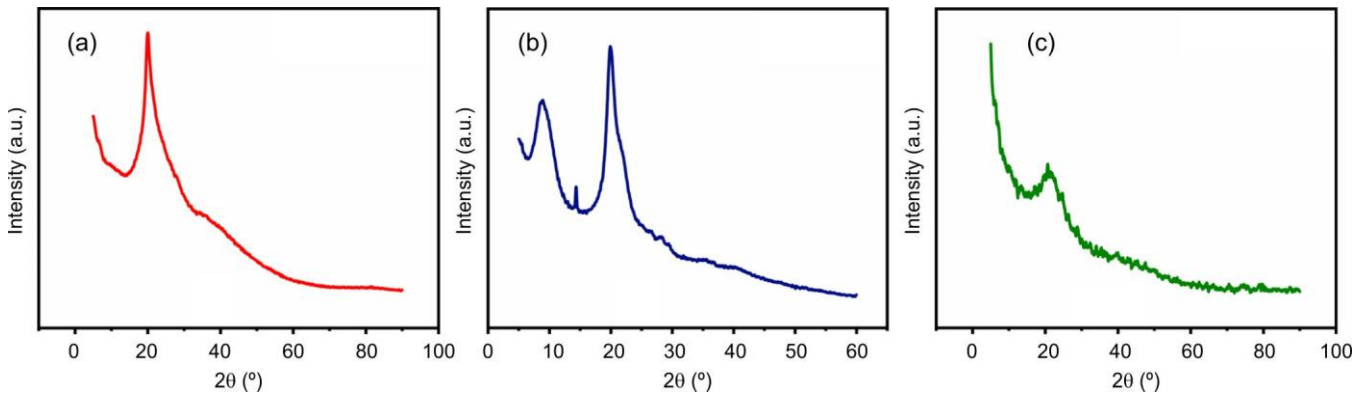


Fig. 2. XRD diffractogram of (a) sodium carboxymethyl cellulose (CMCNa) (b) chitosan (Chit) (c) crosslinked hydrogel film (Chitcmc)

interaction between $–COO^-$ and $–NH_3^+$ thereby indicating the successful formation of the Chitcmc hydrogel through electrostatic interaction [43,44].

Thermal studies: The thermogram of sodium carboxymethyl cellulose (CMCNa), chitosan (Chit) and crosslinked hydrogel film (Chitcmc) are shown in Fig. 3. In the thermo-

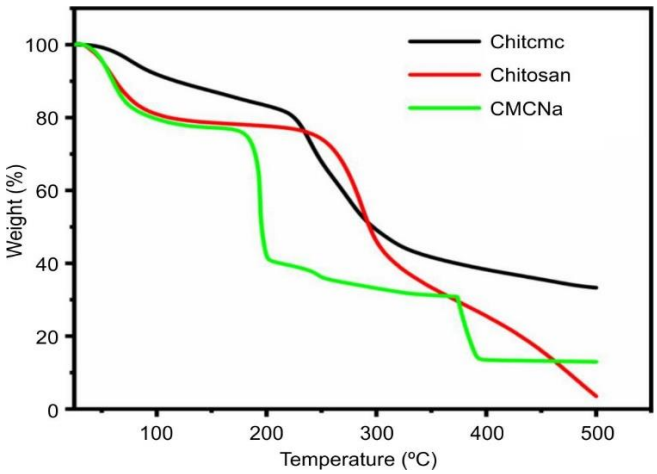


Fig. 3. TGA curves of sodium carboxymethyl cellulose (CMCNa), chitosan (Chit) and crosslinked hydrogel film (Chitcmc)

gram of CMCNa, three distinct decomposition phases were observed in the range of 34.33-167 ºC, 175-371.67 ºC and 371.67-395 ºC with a weight loss of about 23.03%, 45.39% and 17.22%, respectively. The first stage of degradation may be attributed to the loss of water molecules bound to the polymer. The second and third stage of degradation may be attributed to the depolymerization of the polymer molecule with the loss of CO_2 from COO^- groups attached to the polymeric network along the formation of water, CO and CH_4. Chitosan shows two stages of degradation in the range of 34-112.67 ºC and 229-500 ºC with a weight loss of about 19.90% and 73.08%, respectively. The first stage of degradation was due to the elimination of adsorbed/absorbed water from the polysaccharide while the second stage of degradation was due to the entire degradation of the polymer occurring *via* thermo-oxidative process. The major weight loss for Chitcmc was in the range of 210-323 ºC with about 37.77% of weight loss. The hydrogel has intermediate thermal stability between the two components, but characterized by a higher residual weight. Further, TGA besides giving information on the thermal behaviour can be also of diagnostic value. The TGA curve of Chitcmc which is different from those of CMCNa and Chit indicated the formation of a new material [45-49].

Morphological studies: The SEM images of Chitcmc hydrogel film at 10.00 kX and 20.00 kX magnifications are shown in Fig. 4a-b, respectively. The micrograph at lower magnification showed coiled structure consisting of interlinked small beads. However, micrograph at higher magnification revealed presence of pores in the backdrop of the coiled beady structure [50]. The EDS spectrum shown in Fig. 4c indicated the presence of the elements C, O, N and Na in the Chitcmc hydrogel.

Swelling studies of hydrogels: The variation of swelling index with respect to change in pH and time is presented in Fig. 5. The swelling of the hydrogel film increased abruptly within first 30 min with the values of 126.5%, 130% and 169.4% at pH 4, 7 and 9.2, respectively. The swelling index values after 24 h of immersion reached about 153.0%, 212.5% and 1547.2% at pH 4, 7 and 9.2, respectively. From the swelling study, it was observed that the swelling index values are dependent on the pH of the medium and increased considerably with increase in time for the three corresponding pH buffer solutions.

The Chitcmc hydrogel film with Chit/CMCNa in the ratio 1:3 has higher CMCNa content. At lower pH, the swelling index value was considerably low compared to the swelling at neutral and basic medium. At pH 4, the amino groups on Chit and the carboxyl groups on CMCNa gets protonated. Although the affinity of NH_3^+ on chitosan to water molecules plays a crucial role in swelling, but owing to the increasing content of CMCNa, the protonated carboxyl groups leads to lowering of Donnan Type effect on the hydrogel by decreasing the negative charges on the polymer chain. Consequently, the swelling index value at pH 4 was observed to be lower compared to at pH 7 and 9.2. In neutral medium, most of the carboxylic group tends to exist as $-COO^-$ leading to electrostatic repulsion between these negative charges, thereby an increase in swelling of the hydrogel was observed compared to the swelling in acidic medium but was lower than that in basic medium. Under alkaline condition, the swelling behaviour was enhanced with increasing content of CMCNa. At pH 9.2, protonation of the amino groups attached to chitosan, could be neglected, wherein most of the amino groups existed as $-NH_2$. However, at higher pH, the carboxylic groups tend

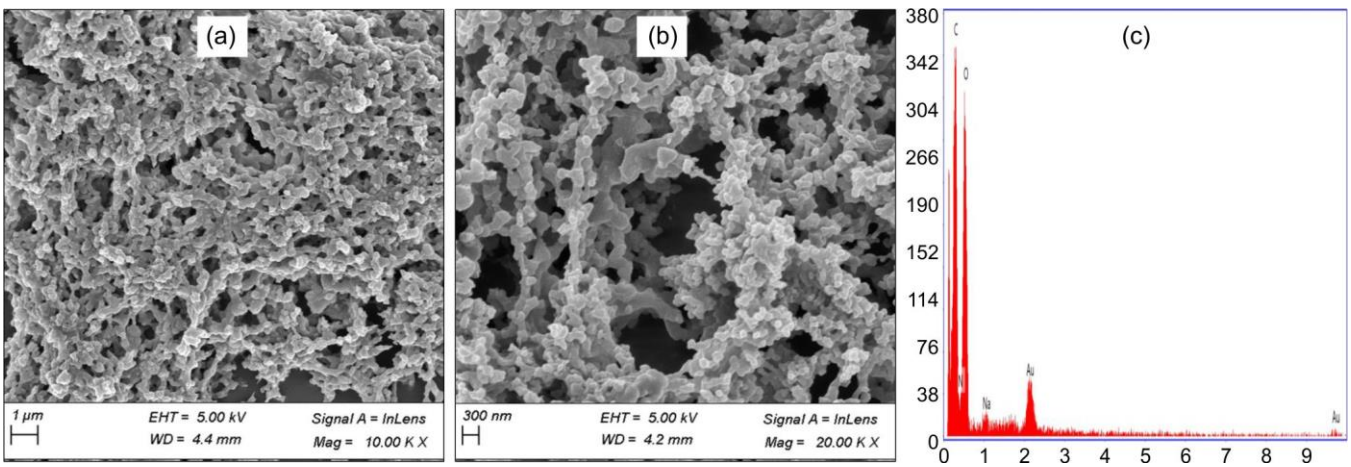


Fig. 4. (a-b) SEM images of Chitcmc hydrogel film at × 10.00 KX and × 20.00 KX magnifications (c) EDS spectrum of Chitcmc hydrogel film

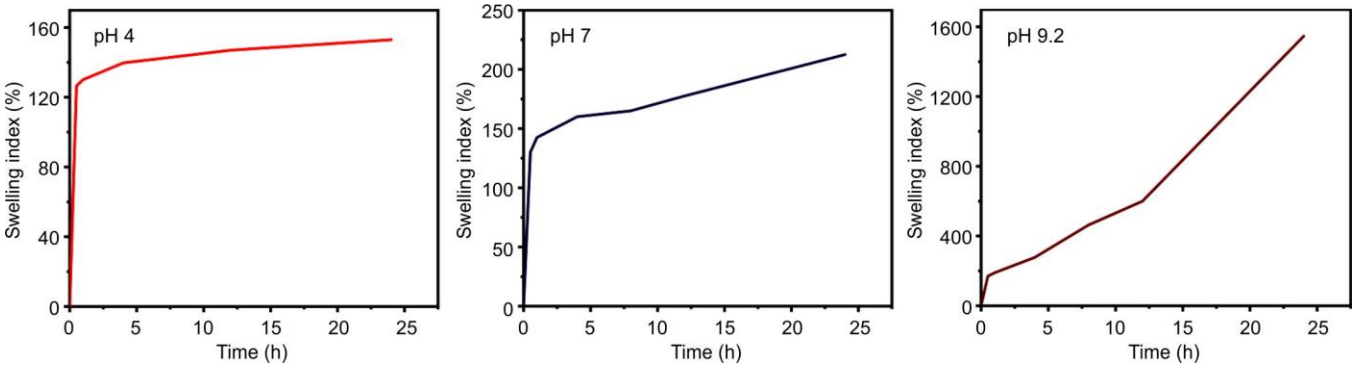


Fig. 5. Swelling profile of Chitcmc hydrogel film at pH = 4, pH = 7 and pH = 9.2

to exist as $–COO^-$ which creates electrostatic repulsion in between the molecular chains which in turn significantly elongates the polymeric chain enhancing higher swelling by the hydrogel [51,52].

Soil pH determination: The pH of a soil indicates the measure of its acidity or alkalinity which has direct impact on the availability of nutrients to plants, on the microbial activity and even on the soil texture. However, the pH range of 6.0-7.0 is considered as the optimal pH range for nutrient availability, microbial activity and also soil texture. Lower pH leads to reduced soil structure, thereby leading to soil erosion and reduced water holding capacity. Similarly, a higher pH value of the soil may also lead to reduced soil structure, thereby leading to soil erosion and reduced water infiltration. Thus, it is noteworthy to mention that when the soil pH is too low or too high, the soil structure may be compromised, consequently leading to soil erosion and reduced water-holding capacity [53]. The pH of soil plays a vital role in reflecting the soil characteristics and is considered as one of the most useful indicators for soil functioning and processes. Soil pH also influences various factors including soil fertility, biogeochemical cycling, bacterial community composition, *etc.* [54,55]. From the experimental analysis, the pH values obtained for the districts Kamrup (M), Kamrup (R), Nagaon, Nalbari and Bajali district of Assam, India were 5.1, 4.2, 6.0, 6.2 and 4.9, respectively.

Grain size analysis: The pipette withdrawal time was calculated using Stokes law after Carver [32] and the data obtained after evaluation of the eight samples have been tabulated in Table-1 while Table-2 displays the grain size parameters graphic mean grain size (M_z), graphic standard deviation (σ_I), graphical inclusive skewness (S_{ki}) and graphical kurtosis (K_G) obtained from the evaluation of grain size analysis.

TABLE-2
GRAIN SIZE PARAMETERS AFTER FOLK AND WARD [30]

Parameters	Formula	Classification
Graphic mean	$M_Z = \frac{\varphi_{16} + \varphi_{50} + \varphi_{84}}{3}$	(-1) – 0 Very Coarse Sand 0 – 1 Coarse Sand 1 – 2 Medium Sand 2 – 3 Fine Sand 3 – 4 Very Fine Sand 4 – 8 Slit >8 Clay
Graphic standard deviation	$\sigma_1 = \frac{\varphi_{84} - \varphi_{16}}{4} + \frac{\varphi_{95} - \varphi_5}{6.6}$	<0.35 Very Well Sorted 0.35 – 0.50 Well Sorted 0.50 – 0.70 Moderately Well Sorted 0.70 – 1.0 Moderately Sorted 1.0 – 2.0 Poorly Sorted 2.0 – 4.0 Very Poorly Sorted > 4.0 Extremely Poorly Sorted
Graphic Skewness	$S_{ki} = \frac{\varphi_{16} + \varphi_{84} - 2\varphi_{50}}{2(\varphi_{84} - \varphi_{16})} + \frac{\varphi_5 + \varphi_{95} - 2\varphi_{50}}{2(\varphi_{95} - \varphi_5)}$	1.0 – 0.30 Very Fine Skewed 0.30 – 0.1 Fine Skewed 0.1 – (-0.1) Near Symmetrical (-0.1) – (-0.3) Coarse Skewed (-0.3) – (-1.0) Very Coarse Skewed
Graphic Kurtosis	$K_G = \frac{\varphi_{95} - \varphi_5}{2.44(\varphi_{75} - \varphi_{25})}$	< 0.67 Very Platykurtic 0.67 – 0.90 Platykurtic 0.90 – 1.11 Mesokurtic 1.11 – 1.50 Leptokurtic 1.50 – 3.00 Very Leptokurtic > 3.00 Extremely Leptokurtic

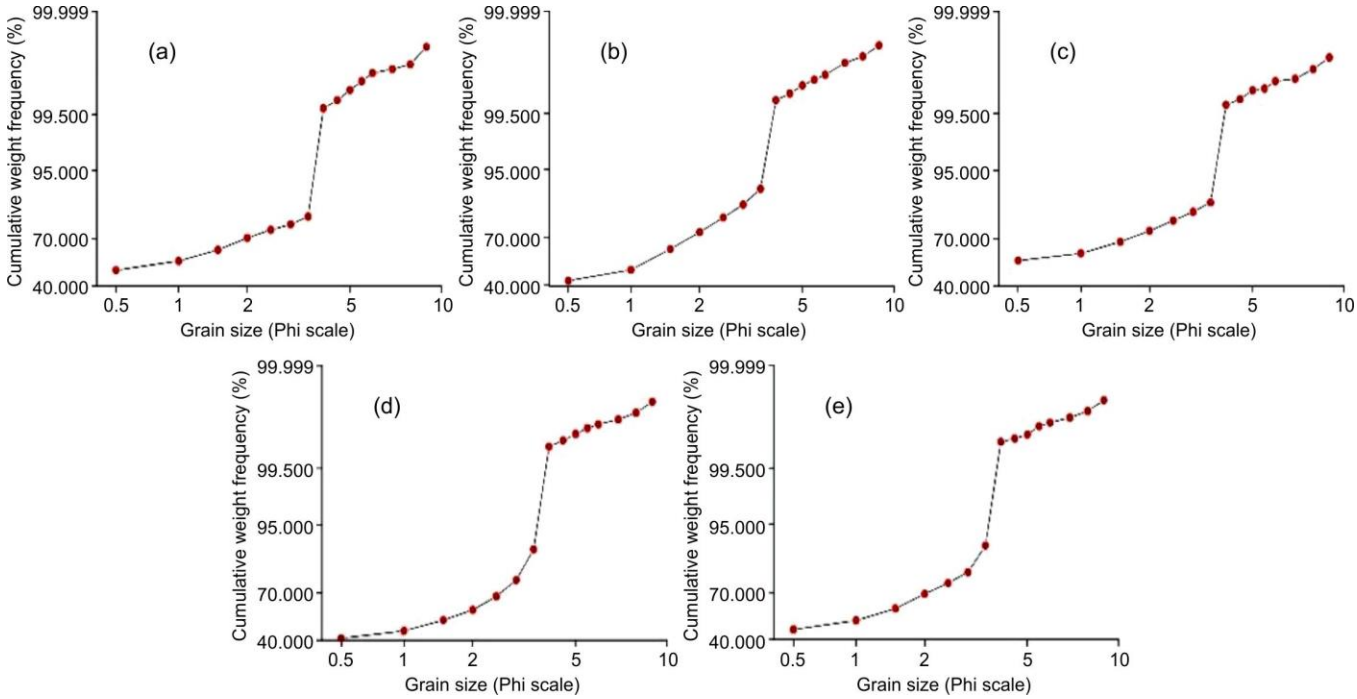


Fig. 6. Arithmetic probability graph prepared by using cumulative weight frequencies of the grains sizes *viz.* (a) Kamrup Rural District, (b) Kamrup Metro District, (c) Nagaon District, (d) Bajali District and (e) Nalbari District

TABLE-3
DATA OF DIFFERENT GRAIN SIZE PARAMETERS FROM ALL FIVE LOCATIONS

Sample location	Graphic mean		Inclusive graphic skewness		Inclusive standard deviation		Graphic kurtosis	
	Value	Interpretation	Value	Interpretation	Value	Interpretation	Value	Interpretation
Kamrup Rural	1.34	Medium sand	0.73	Very fine skewed	1.44	Poorly sorted	0.61	Very platykurtic
Kamrup Metro	1.34	Medium sand	0.38	Very fine skewed	1.29	Poorly sorted	0.7	Platykurtic
Nagaon	1.10	Medium sand	0.87	Very fine skewed	1.33	Poorly sorted	1.15	Leptokurtic
Bajali	1.53	Medium sand	0.25	Very fine skewed	1.37	Poorly sorted	0.5	Very platykurtic
Nalbari	1.67	Medium sand	0.52	Very fine skewed	1.17	Poorly sorted	0.51	Very platykurtic

The graphic mean (M_z) of Kamrup Metro District, Kamrup Rural District, Nagaon District, Nalbari District and Bajali District are 1.34, 1.34, 1.10, 1.67 and1.53, respectively. The results indicated that all the samples belong to medium sand category. The calculated graphic standard deviation of Kamrup Metro District, Kamrup Rural District, Nagaon District, Nalbari District and Bajali District are 1.29, 1.44, 1.33, 1.17 and 1.37, respectively, which shows that they belong to Poorly Sorted category. Also, the graphic skewness values of Kamrup Metro District, Kamrup Rural District, Nagaon District, Nalbari District and Bajali District are 0.38, 0.73, 0.87, 0.52 and 0.25, respectively, resulted in they belong to very fine skewed category.

Classification of soil texture: The arithmetic probability graph prepared by using cumulative weight frequencies of the grains sizes are displayed in Fig. 6 and the result of grain size parameters of the soil samples collected from five different locations are listed in Table-3.

Moreover, the Graphic Kurtosis of Kamrup Metro district, Kamrup rural district, Nagaon district, Nalbari district and Bajali district are 0.70, 0.61, 1.15, 0.51 and 0.50. The result shows that Kamrup Rural, Nalbari and Bajali belong to Very Platykurtic category, Kamrup Metro under Platykurtic and Nagaon under Leptokurtic category. The textural classification of the soil samples shows that Kamrup Metro, Nagaon, Nalbari and Bajali falls under the sand category and Kamrup Rural under the loamy sand category as represented in Fig. 7.

Water holding capacity of hydrogel in soil: Water-holding substances can enhance soil moisture, limit the rate of evaporation of soil water or even provide solution to the environmental problems faced by the arid areas due to drought

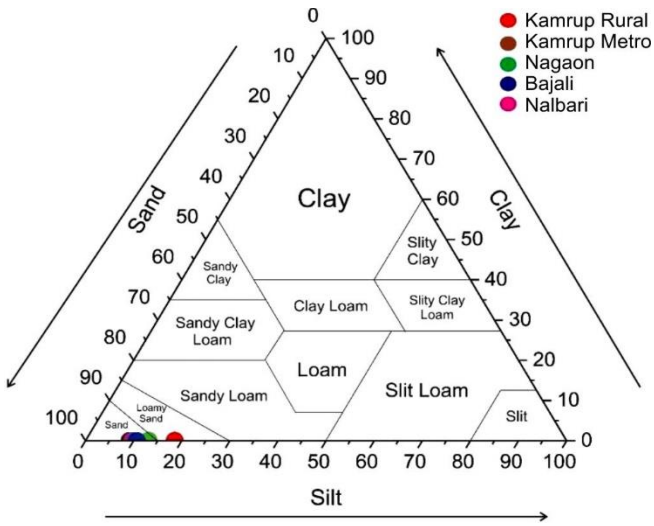


Fig. 7. Textural classification of soil after USDA, 2017 [56]

or water shortage [57]. Most often, the hydrogels are used as soil conditioners that can considerably enhance the growth of plants by providing them with periodic nutrients and water content. These soil hydrogels can boost up a nutrient rich environment, maximizing water absorbency and ensuring slow release of the nutrients [58]. In this work, potentiality of the Chitcmc hydrogel for practical application in agriculture was studied by evaluating the water holding capacity of the hydrogel sample in the sandy soil samples collected from the five districts. The values of water holding for the soil samples collected from the five districts of Assam state, India, have been tabulated in Table-4. The variation in water holding capacity with respect to soil samples and amount of hydrogel addition is shown in Fig. 8.

Effect of soil pH on water holding capacity (WHC): The pH of a soil indicates the measure of its acidity or alkalinity which has direct impact on the availability of nutrients to plants, on the microbial activity and even on the soil texture. However, the pH range of 6.0-7.0 is considered as the optimal pH range for nutrient availability, microbial activity and also soil texture. Lower pH leads to reduced soil structure, thereby leading to soil erosion and reduced water holding capacity. Similarly, a higher pH value of the soil may also lead to reduced soil structure, thereby leading to soil erosion and reduced water infiltration. Thus, it is noteworthy to mention that when the soil pH is too low or too high, the soil structure may be compromised, consequently leading to soil erosion and reduced water-holding capacity [53]. From the experimental analysis, WHC was found to be higher for the untreated soil samples collected from Nagaon (54.62%) and Nalbari (50.66%) having pH in the range of 6.0-7.0, respectively. However, lower values of water holding capacities were observed for the untreated sandy soil samples collected from Kamrup (M), Kamrup (R) and Bajali districts of Assam, India, which may be attributed to lower pH value for the corresponding soil samples.

TABLE-4
PERCENTAGE OF WATER HOLDING

District	pH	Type of soil	Water holding capacity (%)			
			Blank soil	Amount of hydrogel added to the soil		
				0.3% (w/w)	0.4% (w/w)	0.5% (w/w)
Kamrup (M)	5.1	Sand	36.89	48.16	50.73	53.44
Kamrup (R)	4.2	Loamy sand	42.74	45.67	47.78	49.07
Nagaon	6.0	Sand	54.62	55.38	57.29	58.98
Nalbari	6.2	Sand	50.66	51.37	51.99	52.31
Bajali	4.9	Sand	49.47	51.52	52.34	53.99

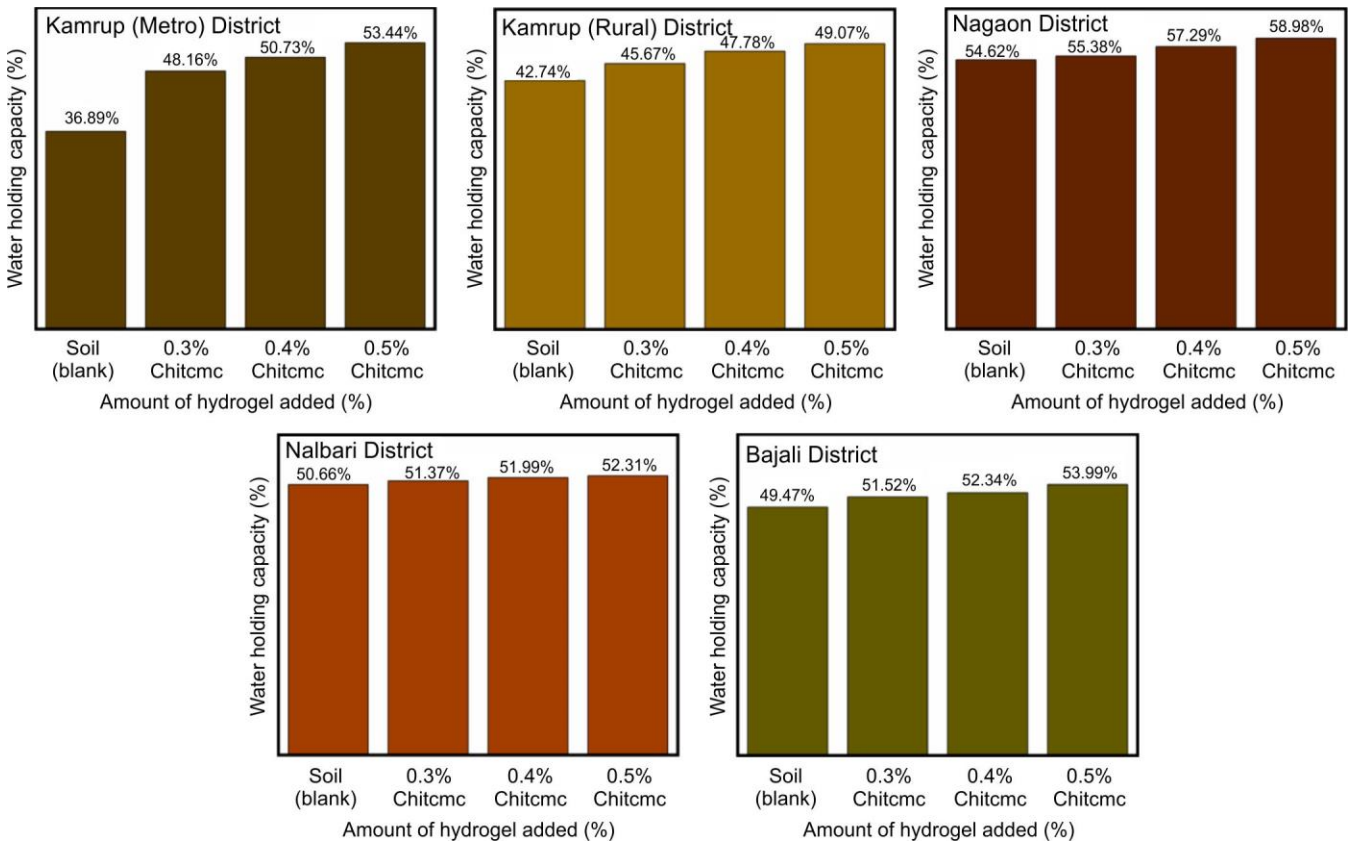


Fig. 8. Water holding capacity by Chitcmc hydrogel sample

Effect of soil texture on water holding capacity (WHC): Soil organic matter and soil texture are the two major factors that influence soil water holding capacity. Higher the organic matter content in soil, greater will be the water holding capacity. Recent studies have established a significant positive relationship between soil water holding capacity with that of the organic matter and clay content of soil and at the same time a negative relationship was established between soil water holding capacity and sand content [59]. Since sandy soils have low water holding capacity (WHC), application of superabsorbent polymers (SAPs) or hydrogels can be effective in increasing its WHC in such soil. For instance, when the hydrogel-amended soil dries, the amount of water absorbed by the hydrogel (about 90-95% of the retained water) can be gradually released to the plants. Therefore, on application of hydrogel, the WHC (amount of water that a soil can hold) as well as the AWC (amount of water that a plant can uptake) of sandy soils can be considerably increased reducing water loss by deep percolation and also fertilizer leaching [60]. From the results, it was observed that although WHC for the soil sample collected from Kamrup (M) district showed the lowest value of WHC (36.89%) in blank soil, but addition of hydrogel with the concentration of 0.3, 0.4 and 0.5%(w/w) increased the WHC in soil by 11.27%, 13.84% and 16.55%, respectively whereas such increase was not observed for all the other sandy soil samples. The reason may be attributed to the proper functioning of the hydrogel in coarse-textured soil as was obtained for the sandy soil sample collected from Kamrup (M) district. The functioning of hydrogel was however hindered in the soil samples collected from Kamrup (Rural), Nagaon, Nalbari and Bajali districts of Assam, India which was found to have fine-textured soil. Accordingly, the percentage of increase in WHC was also not very prominent for the soil samples collected from these four districts even on addition of hydrogel. The present findings are in conformity with previously reported work by Albalasmeh *et al.* [61]. It was reported that hydrogel application was more effective in coarse-textured soil compared to fine-textured soil. Moreover, increasing the hydrogel concentration also resulted in enhancement of soil physical and hydraulic properties. Water use efficiency was reported to increase from 13% to 41% for sandy soil at a hydrogel concentration of 1%.

Effect of hydrogel addition on water holding capacity (WHC): As can be seen from Table-4, irrespective of the soil texture, WHC for all the sandy soil samples increased significantly on addition of hydrogel compared to the WHC of the untreated soil. Moreover, the percentage of water holding values was also found to increase with increase in the amount of hydrogel addition from 0.3-0.5% (w/w) to soil. All the soil samples followed an identical trend of increase in water holding capacities with increase in percentage of hydrogel addition. Similar results were reported by Huttermann *et al.* [62] wherein sandy soil sample was treated with superabsorbent hydrogel concentrations of 0.04%, 0.08%, 0.12%, 0.20% and 0.40% and it was observed that water retention capacity of the soil increased exponentially with increase in hydrogel additions to the soil. Koupai *et al.* [63] reported that amongst the application of 2, 4, 6 and 8 g/kg of hydrogel samples addition to sandy loam soil, significant enhancement of the available water content approximately by 3-folds was observed for the soil sample treated with 8 g/kg of hydrogel addition to the soil compared to that of the control. Naushabayev *et al.* [64] also reported that with respect to the untreated soil, the application of hydrogel can be effective in increasing the moisture content in sandy soils. Similar results were also reported by Agaba *et al.* [65] wherein 0.4% hydrogel amendment was found to be more effective in retaining water compared to 0.2% or control in the same sandy soil. The results therefore indicated that with increase in hydrogel concentration, the water use efficiency can be simultaneously enhanced. Application of hydrogel to sandy soils can enhance the amount of moisture content around the root zone by increasing the retention pores and reduce saturated hydraulic conductivity by decreasing the drainage pores. Therefore, application of hydrogels to sandy soils can lead to significant increase in water retention capacity [66].

Conclusion

A mixed hydrogel based on chitosan/sodium carboxymethyl cellulose in presence of malonic acid as crosslinker was synthesized and characterized. The swelling study was carried out at pH 4, 7 and 9.2 and the swelling index values obtained after 24 h of immersion of the hydrogel sample were 153.0%, 212.5% and 1547.2%, respectively. The maximum swelling capacity of the hydrogel was observed in alkaline medium making it suitable for application in saline-alkali soils. The percentage of water holding investigated on blank soil, collected from Kamrup (M), Kamrup (R), Nagaon, Nalbari and Bajali districts, were found to be 36.89%, 42.74%, 54.62, 50.66% and 49.47%, respectively. As soil with pH range 6.0-7.0 shows greater WHC, the soil samples from Nagaon (pH ~ 6) and Nalbari (pH ~ 6.2) showed maximum WHC. With addition of 0.3% (w/w), 0.4% (w/w) and 0.5% (w/w) of hydrogel to the soil samples (10 g), WHC were found to increase for all the treated soil samples with increase in the concentration of the hydrogel addition to soil. However, the textural analysis of the soil samples indicated close similarity though collected from different districts. This is the reason why there is not very prominent variation in water holding capacity for the collected soil samples. Nevertheless, the Chit-CMC hydrogel was found to be more effective in the sandy soil with a coarse texture, as observed in the sample collected from Kamrup (M), which was relatively more coarse-textured. Further work using prepared soil samples with known textural variations will be necessary to better understand the relationship between WHC and soil texture, as well as the specific role of hydrogels in soil systems.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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