Journal of Pharmacognosy and Phytochemistry
Available online at www.phytojournal.com

E-ISSN: 2278-4136
P-ISSN: 2349-8234
JPP 2017; SP1: 42-48

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Characterization of water retention and release capacity of innovative nano clay polymer composite superabsorbent

Mahendra Kumar Verma, Priyal Pandey and Nirmal De

Abstract
Use of innovative nano clay polymer composite (NCPC) superabsorbent was explored particularly to ameliorate moisture stress condition under rainfed ecosystem. NCPC were synthesized by polymerization reaction with 10% acrylic acid, acrylamide, 0.9% ammonium per sulphate as initiator, 0.12% N, N′-methylene bis acrylamide loaded with 10% kaolinite clay at 65^0C reaction temperature in presence of inert nitrogen gas. The water sorption was found maximum (131.25 g H_2O/g NCPC) when kaolinite clay was modified by hot water FYM extract irrespective of amount of loaded phosphorus in NCPC (treatment 15 & 16) as compared to pure clay 90.04 g H_2O/g NCPC without any modification (T14). The average water desorption pattern was also found to be conducive when clay was modified by hot water FYM extract. The rate kinetics for water sorption and desorption was studied under different innovative modified NCPC. The present study revealed that when NCPC added in alluvial soil it helps to maintain soil moisture regime for additional 8-13 days more (depending on composition) as compared to no application of NCPC in soil. Superabsorbent polymeric materials in combination with modified clay play a significant role in improving and retaining soil moisture regime and further reducing the rate of desorption of water act as an effective material for higher water use for agricultural applications.

Keywords: NCPC as a Superabsorbent, Water Absorbency, Equilibrium Water Absorbency, Average Water desorption pattern and Water Retention capacity.

Introduction
Superabsorbent polymers were first developed in 1970s by USDA, for improving the water holding capacity of soils, to promote seed germination and plant growth (Liu and Guo, 2001) [9]. Superabsorbent polymers (SAP) are three-dimensional cross-linked hydrophilic materials that can absorb and retain large quantities of water or aqueous solutions. Superabsorbent are hydrophilic polymers in a three dimensional network (SAPs) (Talaat *et al*., 2008) [18]. Acquired superabsorbent resin can absorb aqueous solution hundreds of times than their own dry sample (Nge *et al*., 2004) [15]. The Hydrogel are known by their ability to absorb and retain large quantities of liquids (swelling) much greater, in terms of weight, than the initial weight of the material (Horie *et al*., 2004) [4]. Superabsorbent can imbibe a large amount of water or aqueous solution and display a slower water release rate. Owing to their excellent properties, superabsorbent are widely used in many fields such as agriculture, bio-medicals, tissue engineering, waste-water treatment and other environmental & chemical engineering fields (Ma *et al*., 2015) [12]. As super absorbent polymer can reduce irrigation water consumption, improve fertilizer efficiency in soil, lower the death rate of plants, and increase plant growth, recently the use of super absorbent polymer has attracted great attention, as water managing material for the renewal of arid and desert environment (Nge *et al*., 2004; Lokhande and Varadara, 1992) [15, 11]. Superabsorbent polymeric materials in combination with clay minerals play a significant role in improving the water-holding capacity (Mortland, 1970) [13]. (Iza *et al*., 1998) [5] reported that the presence of kaolin and poly (acrylic acid-co-acrylamide) content significantly affects the water absorbency and diffusion coefficient of urea released from poly-(AA-coAm)/kaolin composite hydrogel. Superabsorbents have shown some encouraging effects in modern agriculture and have exhibited great potential and perspective. On the basis of our previous work about a superabsorbent containing Kaolinite clay, in this study The P coated nano clay polymer composite were developed through polymerization reaction and effect of nano clay polymer composite as superabsorbent on the Water Absorbency investigated. Average Water desorption pattern and Water Retention capacity were also evaluated.

Materials and Methods

Preparation of Nano clay polymer composite

According to Ling and Liu, (2007) [8] standard procedure preparation of NCPC is as follow: The P coated nano clay polymers composite were developed in the soil physics lab, Department of soil science & agricultural chemistry, Institute of agricultural sciences, BHU, Varanasi during 2014-2015. Nano clay polymer composite (NCPC) super absorbent were synthesized by polymerization reaction with 10% acrylic acid, acrylamide, 0.9% ammonium per sulphate as initiator, 0.12% N,N'- methylene bis acrylamide as cross linker loaded with 10% Kaolinite clay at 65^0C reaction temperature in presence of nitrogen gas. Just before polymerization reaction 10%, 15% and 20% potassium dihydrogen phosphate (KH_2PO_4) was added which lead to synthesis of P coated nano clay polymer composite. The dried at 100^0C temperature to a constant weight. Finally, drying, grinding and screening of samples were done. This sample product was tested for water sorption properties and retention capacity.

Table1: Treatment details

T_{12}	Pure Kaolinite+ NCPC with P @ 10%
T_{13}	Pure Kaolinite+NCPC with P @ 15%
T_{14}	Pure Kaolinite NCPC with P @ 20%
T_{15}	Hot water FYM extract modified Kaolinite + NCPC with P @ 10%
T_{16}	Hot water FYM extract modified Kaolinite + NCPC with P @ 15%
T_{17}	Hot water FYM extract modified Kaolinite + NCPC with P @ 20%
T_{18}	CTAB modified Kaolinite + NCPC with P @ 10%
T_{19}	CTAB modified Kaolinite + NCPC with P @ 15%
T_{20}	CTAB modified Kaolinite + NCPC with P @ 20%

Measurement of water absorbency of NCPC

A series of small tea bag containing an accurate dry weight of finely powdered P coated NCPC samples (0.1g) were immersed in distilled water, at room temperature. The Swollen samples were then separated from unabsorbed water by filtering and hung up for 15 min. until no liquid was dropped off from the sample. The water absorbency, Q of sample was calculated according to following equation;

Q= (M2-M1)/M1

Where, M1 and M2 are the weight of dry sample and swollen sample, respectively. Q was calculated as grams of water per gram dry hydrogel (g/g).

Measurement of equilibrium water absorbency of NCPC in distilled water

Series of small tea bag containing an accurate dry weight of finely powdered NCBPC samples (0.1g) were immersed in distilled water, at room temperature. Swollen samples were weighted at different time interval up to it reached a constant Weight. The Swollen samples separated from water at different time by filtering and hung up for 15 min. until no liquid was dropped off from the sample. The water absorbency, Q of sample was calculated according to following equation;

Q= (M2-M1)/M1+T

Where, M1 and M2 are the weight of dry sample and swollen sample and T are taking weight different time interval respectively. Q was calculated as grams of water per gram dry hydrogel (g/g).

Measurement of Water Retention in soil

For measurement of water retention percent we take a pot which was perforated at the base and filled it with 50gm soil and add 0.1gm NCPC. After then put it in a water tub so the soil reached at saturation. Then remove the pot from water tub. After then weight it. Now we take weight of this soil every day up to it reach their initial. Then using the following formula we calculated the water retention percent of it. Formula is:

WR% = Wt-Wi/Weq-Wi×100

Where, WR% = percent water retain.

Wt. = weight at different time.

Wi = initial weight.

Weq.= equilibrium weight.

Statistical analysis

The data were subjected to one way analysis of variance (ANOVA) using SPSS version 23 software. Duncan's multiple range test (DMRT) was performed to test the significance of difference between the treatments.

Results and Discussion

Water absorbency and equilibrium water absorbency of NCPC in distilled water

Water absorbency and equilibrium water absorbency as influenced by different treatment were presented in the table 2, 3 and figure1, 2. The absorbency found maximum in T15 (Hot water FYM extract modified kaolinite +NCPC with P @ 10%) was 131.25 g H_2O/g NCPC with the CD $_{0.05}$=2.032. Followed by T16 (Hot water FYM extract modified Kaolinite + NCPC with P @ 15%) was 128.16 g H_2O/g NCPC and T17 (Hot water FYM extract modified Kaolinite + NCPC with P @ 20%) was126.09 g H_2O/g NCPC. The minimum found in T14 (Pure Kaolinite NCPC with P @ 20%) was 90.04 g H_2O/g NCPC. All treatment performs better than pure Kaolinite +NCPC. It also found that initially the rate of water absorbency was very fast than it decline. The swelling rate of superabsorbent composites was mainly influenced by the swelling ability, the particle size, the surface area as well as the density of the superabsorbent (Pourjavadi and Salimi, 2008) [16]. During the initial 50 min, the swelling rate was higher than that of later swelling process. As the swelling process continuing, the swelling rate was reduced, and the curves of the swelling rate became flatter and swelling equilibrium could be finished within 60 minutes then after decline. Similar result obtained by (Mukhopadhyay, 2014) [14] the increase in water absorbency of NCBPC with increasing time; it was minimum at 10 minutes only 23 mL and maximum within 60 minutes i.e. 144.6 mL of water from 1g of NCPC. According to (Wu *et al*., 2000) [20] the *Q*H2O varies according to time and reached highest to 4000 g H2O within 60 minutes for Starch-graft polyacrylamide/ Kaolinite composite. Within 50 min T15 swell 88.26% and all other treatments also show same trend. So we concluded that due to modification of clay it increase the water absorbency of NCPC. The physical filling of kaolin particle in the polymer decreases the amount of hydrophilic groups and consequently results in the reduction of water absorbency (Lee *et al*., 2004) [7]. According to (Liang and Liu 2007) [8], the changes in the water absorbency of the product is due to existence of synergistic effect among the groups of –COOH, –COO–, –CONH2 and –OH (on kaolin). (Guo *et al*., 2005, Sarkar *et al*., 2013 and Jatav *et al*., 2013) [3, 17, 6]. Are reported that the high water absorbency with time is probably due to additional polymeric network and hydrophilic interaction between clay and polymer increase the network to hold a large amount of water. Loaded of P @ 10%, 15% and 20% of NCPC during polymerization reaction. Treatments 10% P have high water absorbency compare than Treatments 20% P loaded, because 20% P more concentration of solution so decrease water absorbency of treatments. The swelling and shrinkage behaviour of the developed NCPC superabsorbent was greatly influence by the characteristic of external solution such as charge valences and salt concentration. The osmotic pressure difference was most important factor. The increasing osmotic pressure of external solution with the increasing concentration of the NaCl solution led to the reduction of osmotic pressure difference between internal and external of superabsorbents, which lower the swelling rate of NCPC. (Wang and Wang, 2009) [19]. Reported that the water absorbency of the superabsorbent decreases with the concentration of the NaCl solution increasing. The osmotic pressure difference between the gel network and external solution decreases, and the screening effect of the counter ion (e.g., Na) with respect to negative COO groups is enhanced when the concentration of the saline solution is increased. As a result, the driving force for water diffusing into the superabsorbent is weakened, and then the water absorbency decreases.

Average Water desorption pattern of NCPC

Average Water desorption as influenced by different treatments were presented in the table 4 and figure 3. The Average Water desorption found that the minimum water retain in T14 (Pure Kaolinite NCPC with P @ 20%) was 72.03g H_2O/g NCPC in 8 (hrs) and1.02 g H_2O/g NCPC in168 (hrs) with the CD $_{0.05}$ at 8 (hrs.) and in168 (hrs.) was 2.845 and 1.756 respectively. The maximum water retain found in T15 (Hot water FYM extract modified Kaolinite +NCPC with P @ 10%) was 109.38 g H_2O/g NCPC in 8 (hrs) and 3.56g H_2O/g NCPC in168 (hrs) with the CD $_{0.05}$ at 8 (hrs.) and in168 (hrs.) was 2.845 and 1.756 respectively. After 168 hrs all treatments show the Average Water desorption value 1.00g H_2O/g NCPC in 240 hrs with the CD $_{0.05}$ N.S. So we conclude that the addition of modified Kaolinite clay in NCPC indicate slow release behaviour of water of NCPC to retain water than pure Kaolinite clay in NCPC. Slow release of water and nutrient is a functional property of NCBPC vis-a-vis retention of maximum water as a reservoir (Sarkar *et al*., 2013, Liu *et al*., 2006, Guo *et al*., 2005 and Wu *et al*., 2000) [17, 10, 3, 20].

Water Retention Capacity of NCPC with Alluvial Soil

We conduct the experiment to find out the water retention capacity of NCPC in alluvial. Water Retention Capacity of NCPC as influenced by different treatments were presented in the table 5 and figure 4. In alluvial soil found highest water retention in T15 which retain water up to 13 days, (75% after 1day to 0.1% after 13 day) with the CD0.05 at 1st and 13th day was 6.469 and N.S. respectively followed by T16 (73.0% after 1day to 0.0% after 13 day). The minimum water retention found in T14 (61.3% after 1 day to 0.0% after 12 day) with the CD0.05 at 1st and 12th day was 6.469 and 0.258 respectively. We also found that all treatments show better performance than T14 (Pure Kaolinite NCPC with P @ 20%). So we conclude that the addition of NCPC in soil increase the water retention and the addition of Hot water FYM extract modified Kaolinite clay in NCPC increase the capacity of NCPC to retain water than pure Kaolinite clay. (Bandyopadhyay *et al*., 2011) [1]. Are reported that organic manure can lead to increase the water holding capacity. It is due to addition of NCPC in soil it makes a network structure and bound the soil to make aggregate so soil water retention increase. Super absorbents increase water retention of soil by reducing evaporation rate and holding large amount of water and releasing it slowly. Similar results were found by (Cannazara *et al*., 2014) [2]. According to Jatav *et al*., (2013) [6] the water desorption rate of soil with NCPC was lower compared to the soil. These results showed that the NCPC had excellent water absorbency, water-retention, and moisture preservation capacity. The reason was that the superabsorbent polymer NCPC could absorb and store a large quantity of the water in soil, and allow the water absorbed in it to be slowly released with the decrease of the soil moisture. The swollen NCPC was just like an additional nutrient reservoir for the plant-soil system. Consequently, it prolonged irrigation cycles, reduced irrigation frequencies, and strengthened the ability of plants to tolerate drought stress. Thus it is very much applicable in rainfed condition where uneven distribution of rainfall occurs, it can be serve the water in stress condition, thereby can mitigate drought condition for the second crop like lentil in rainfed rice based ecosystem.

Table 2: Water Absorbency (g H_2O/g NCPC) (mean of 3 replicates ± SE).

Treatment	Water Absorbency (g H_2O/g NCPC)
T12	94.88 ± 0.688^c
T13	92.49 ± 0.779^b
T14	90.04 ± 0.557^a
T15	131.25 ± 0.983^i
T16	128.16 ± 0.502^h
T17	126.09 ± 0.560^g
T18	110.91 ± 0.940^f
T19	108.61 ± 0.323^e
T20	105.71 ± 0.400^d
SE(m) ±	0.672
CD (0.05)	2.032

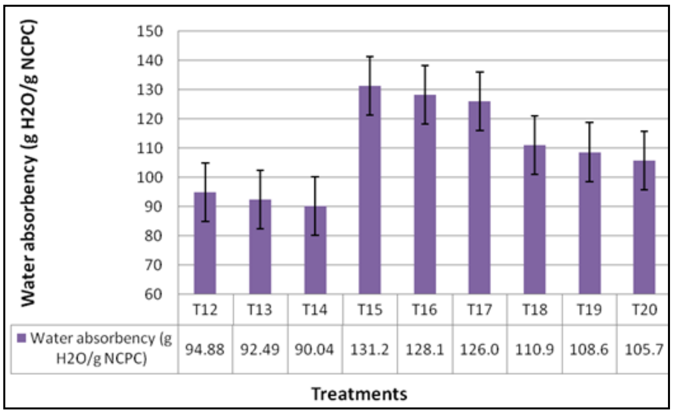

Fig 1: Water Absorbency (g H_2O/g NCPC)

Table 3: Equilibrium Water Absorbency (g H_2O/g NCPC) (mean of 3 replicates ± SE).

Treatments	Equilibrium Water Absorbency (g H_2O/g NCPC) Time (min)							
	10 (min)	20 (min)	30 (min)	40 (min)	50 (min)	60 (min)	8 (hrs)	24 (hrs)
T12	9.63±1.186[ab]	14.64±1.166[ab]	23.64±1.117[a]	51.42±1.064[a]	75.98±0.651[b]	94.88±0.632[b]	94.88±0.061[c]	94.78±1.220[b]
T13	9.6±0.623[ab]	13.73±1.262[ab]	22.73±1.322[a]	49.96±0.115[a]	74.19±1.230[ab]	92.49±1.162[ab]	92.49±0.632[b]	92.37±0.506[ab]
T14	7.24±1.185[a]	12.35±0.686[a]	21.24±1.214[a]	48.47±0.490[a]	72.14±1.160[a]	90.04±1.209[a]	90.04±0.060[a]	89.89±0.080[a]
T15	13.24±1.763[c]	21.82±0.686[d]	41.16±1.279[e]	73.61±1.642[d]	112.48±1.610[f]	131.25±1.787[f]	131.25±1.162[g]	131.19±1.222[f]
T16	10.34±1.161[abc]	17.91±0.686[c]	37.03±0.745[d]	69.48±1.641[c]	108.35±1.227[e]	128.16±1.781[ef]	128.16±1.204[f]	128.09±1.047[e]
T17	8.05±0.615[ab]	15.62±1.167[bc]	34.74±1.279[d]	68.3±0.472[c]	106.28±1.154[e]	126.42±0.940[e]	126.09±1.100[f]	125.99±0.651[e]
T18	11.2±1.187[bc]	15.33±1.263[abc]	30.78±1.215[c]	59.45±1.064[b]	90.11±1.231[d]	110.91±1.209[d]	110.91±0.634[e]	110.85±1.221[d]
T19	9.02±1.161[ab]	14.25±0.595[ab]	29.2±0.706[bc]	58.76±1.064[b]	88.72±1.230[cd]	108.61±1.208[cd]	108.61±0.632[e]	108.52±0.650[d]
T20	7.47±0.546[a]	12.57±0.686[ab]	26.91±0.745[b]	56.47±0.685[b]	85.81±0.653[c]	105.71±1.587[c]	105.71±0.631[d]	105.59±0.645[c]
SE(m) ±	1.175	1.131	1.104	1.028	1.482	1.621	1.042	0.888
CD (0.05)	2.482	2.524	1.478	2.774	3.896	4.739	2.590	2.660

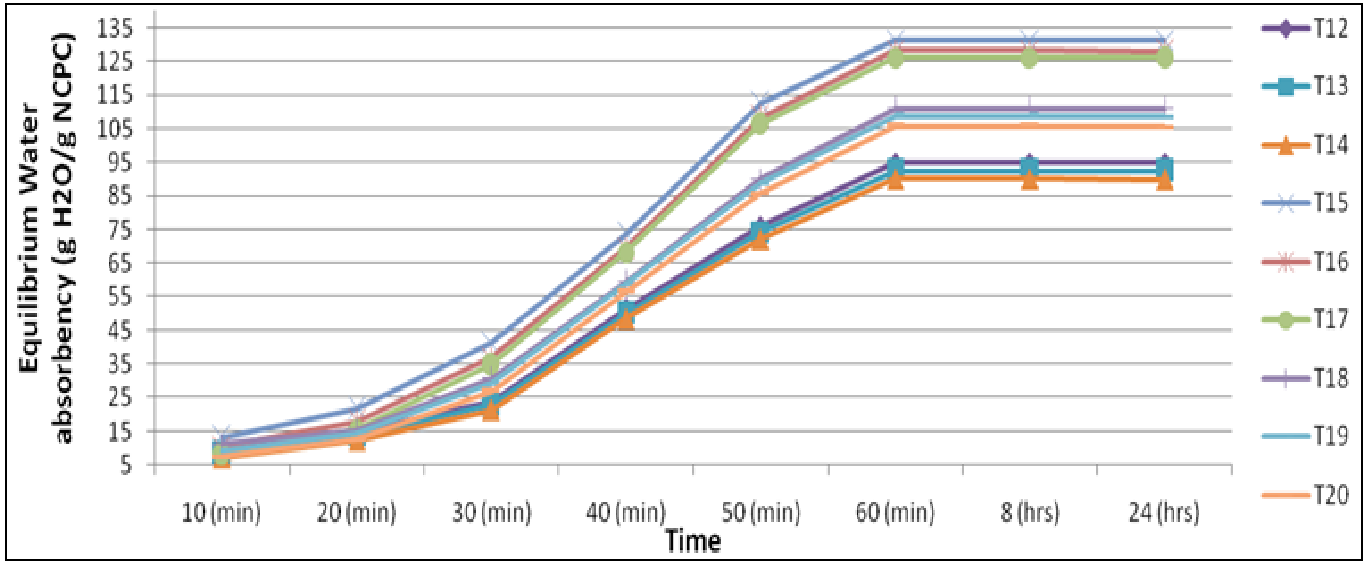

Fig 2: Equilibrium Water Absorbency (g H_2O/g NCPC)

Table 4: Average Water desorption pattern (g H_2O/g NCPC) (mean of 3 replicates ± SE).

Treatments	Average Water desorption pattern (g H_2O/g NCPC)					
	Time (hrs)					
	8 (hrs)	24 (hrs)	72 (hrs)	120 (hrs)	168 (hrs)	240(hrs)
T12	75.90±1.148[a]	56.88±1.727[a]	32.69±1.767[a]	9.89±1.348[b]	1.11±0.103[a]	1.00±0.000[a]
T13	73.99±1.195[a]	54.97±1.195[a]	30.78±1.364[a]	7.98±0.785[ab]	1.08±0.920[a]	1.00±0.000[a]
T14	72.03±1.146[a]	53.01±1.732[a]	28.82±1.341[a]	6.02±0.957[a]	1.02±0.916[a]	1.00±0.000[a]
T15	109.38±2.321[c]	91.58±1.726[c]	61.60±1.463[c]	17.94±1.727[e]	3.56±1.230[b]	1.00±0.000[a]
T16	106.80±1.729[c]	89.00±2.309[c]	59.02±2.091[c]	15.36±0.785[de]	3.19±0.654[b]	1.00±0.000[a]
T17	105.08±1.114[c]	87.28±0.704[c]	57.30±0.376[c]	13.64±1.342[cd]	2.67±0.659[ab]	1.00±0.000[a]
T18	92.43±1.148[b]	74.03±1.160[b]	50.03±1.867[b]	11.27±1.136[bc]	2.98±0.654[ab]	1.00±0.000[a]
T19	90.51±1.472[b]	72.11±1.723[b]	48.44±0.886[b]	9.35±1.138[ab]	2.18±0.504[ab]	1.00±0.000[a]
T20	88.09±1.108[b]	69.69±1.752[b]	46.02±1.132[b]	7.78±0.225[ab]	1.76±0.090[ab]	1.00±0.000[a]
SE(m) ±	1.141	1.525	1.731	1.150	1.027	0.065
CD (0.05)	2.845	3.952	4.832	2.447	1.756	N.S.

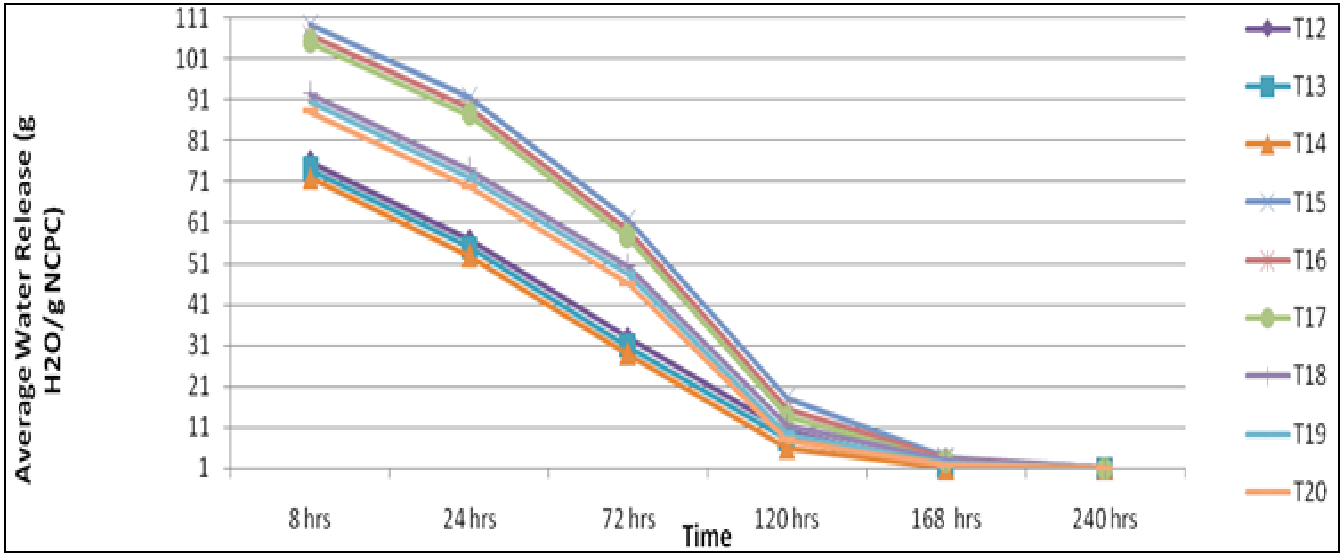

Fig 3: Average Water Release (g H_2O/g NCPC)

Table 5: Water Retention Capacity of Alluvial Soil with NCPC (mean of 3 replicates ± SE).

Water Retention (%)													
Treatments	1 day	2 day	3 day	4 day	5day	6 day	7 day	8 day	9 day	10 day	11 day	12 day	13 day
T12	64.8±2.608^{abc}	57.2±1.101^{ab}	40.3±2.645^{ab}	42.2±2.498^{abc}	35.3±2.542^{abc}	29.6±1.792^{abc}	22.2±2.081^{ab}	16.1±2.251^{abc}	10±2.081^{ab}	5.8±2.025^{abc}	0.8±0.057^{a}	0.5±0.230^{ab}	0.0±0.000^{a}
T13	62.5±1.752^{ab}	55.5±4.582^{a}	42.6±1.327^{ab}	41.1±1.732^{ab}	35.1±2.281^{ab}	27.4±1.824^{ab}	20.3±1.965^{a}	14.9±2.309^{ab}	9.4±1.858^{ab}	4.7±1.473^{ab}	0.6±0.115^{a}	0.4±0.152^{ab}	0.0±0.000^{a}
T14	61.3±2.872^{a}	54.4±1.824^{a}	37.9±1.732^{a}	37.4±2.306^{a}	32.4±1.928^{a}	25.2±1.418^{a}	18.5±1.652^{a}	12.4±2.402^{a}	7.4±1.803^{a}	2.9±1.000^{a}	0.3±0.115^{a}	0.0±0.000^{a}	0.0±0.000^{a}
T15	75.7±1.792^{e}	65.0±1.732^{c}	57.6±2.107^{d}	50.6±2.804^{d}	42.0±2.645^{c}	35.3±3.043^{c}	29.4±2.946^{c}	25.3±2.977^{d}	18.9±2.886^{c}	14.4±2.500^{d}	4.6±1.914^{c}	1.3±0.404^{b}	0.1±0.057^{b}
T16	73.0±1.154^{de}	64.3±2.400^{bc}	53.0±2.309^{d}	48.8±2.916^{cd}	41.4±1.914^{bc}	34.5±2.370^{bc}	28.4±2.589^{bc}	23.7±2.369^{cd}	16±2.645^{bc}	11.3±1.652^{cd}	3.9±1.154^{bc}	1.0±0.577^{ab}	0.0±0.000^{a}
T17	72.5±2.778^{de}	63.9±2.309^{bc}	53.2±1.703^{d}	47.3±1.569^{bcd}	41.2±2.003^{bc}	34.0±1.732^{bc}	27.6±2.050^{bc}	21±2.309^{bcd}	15.6±2.227^{bc}	10±1.732^{bcd}	3.0±1.154^{abc}	1.0±0.577^{ab}	0.0±0.000^{a}
T18	70.6±1.824^{cde}	64.1±2.042^{bc}	48.8±1.628^{cd}	46.9±2.309^{bcd}	39.3±1.652^{bc}	33.5±2.107^{bc}	27.8±1.703^{bc}	20.1±2.309^{abcd}	14.4±1.858^{abc}	9.6±2.136^{bcd}	2.6±0.901^{abc}	0.9±0.208^{ab}	0.0±0.000^{a}
T19	69.2±2.303^{bcde}	58.2±0.577^{abc}	46.9±1.587^{bc}	45.0±1.732^{bcd}	37.3±1.216^{abc}	30.9±2.081^{abc}	25.5±1.847^{abc}	18.8±2.122^{abcd}	12.7±2.193^{abc}	8.1±1.484^{abc}	2.1±0.519^{abc}	0.7±1.000^{ab}	0.0±0.000^{a}
T20	66.2±1.726^{abcd}	57.5±1.587^{abc}	41.3±2.400^{abc}	43.1±1.761^{abc}	35.9±1.732^{abc}	30.1±2.608^{abc}	23.3±2.136^{abc}	16.9±1.732^{abc}	10.4±1.928^{ab}	6.7±1.677^{abc}	1.0±0.577^{ab}	0.5±0.000^{a}	0.0±0.000^{a}
SE(m) ±	2.160	2.277	1.983	2.232	2.035	2.161	2.145	2.329	2.194	1.790	0.930	0.119	0.019
CD(0.05)	6.469	6.818	5.937	6.682	6.093	6.470	6.421	6.974	6.569	5.360	2.783	0.258	N.S.

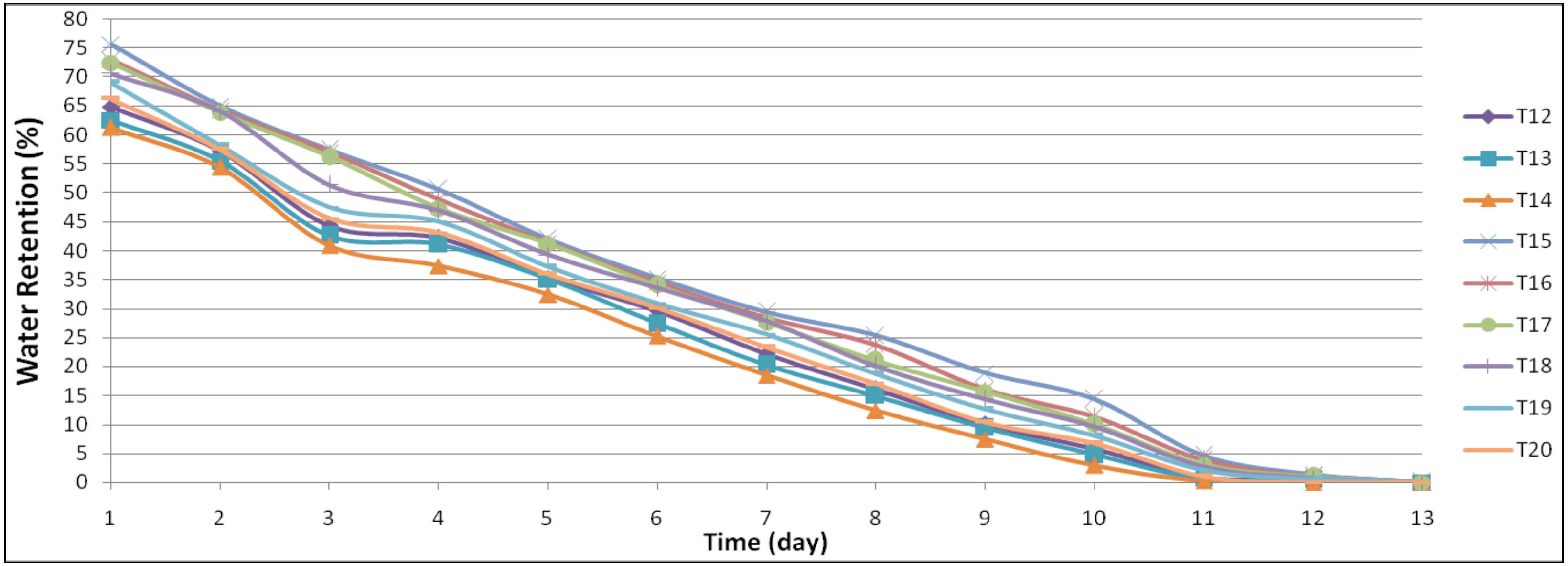

Fig 4: Water Retention Capacity of Alluvial Soil with NCPC

Conclusion

The experiment showed that synthesis of NCPC with modified clay has high water absorbency, slow release of water and high water retention capacity than NCPC with pure clay. NCPC as a superabsorbent improved water-absorption and water-retention capabilities and swelling rates. The P coated nano clay polymers composite as a superabsorbent is a suitable candidate for applications in rainfed agriculture and mitigate to water stress condition.

Acknowledgement

We sincerely acknowledge the technical help and guidance of Dr. S.C. Datta, Principal Scientist, Indian Agricultural Research Institute, New Delhi. The senior author is indebted to the University Grant Commission, New Delhi, for granting funds in a major research project on nano technology in agriculture (BHU project code-P-01/683).

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