Micro encapsulation and characterization of Diclosulam in Xanthan Gum based polymeric system for smart delivery of herbicide in crop production

.

ABSTRACT

Aim: Micro encapsulation of diclosulam herbicide was done in xanthan gum based polymeric system through ionotropic gelation method to formulate a slow-release herbicide to achieve prolonged weed control in irrigated upland ecosystem.

Place and duration of study: The slow-release formulation of diclosulam was synthesized and characterized in the Department of Nano Science & Technology, Tamil Nadu Agricultural University during January to July 2022.

Methodology: Xanthan gum- alginate microsphere system was synthesized with varying concentrations of calcium chloride ($CaCl_2$) (2, 4 and 6 per cent) to encapsulate diclosulam through ionotropic gelation. The size, entrapment efficiency, pore volume, pore radius, surface area and swelling behavior of microspheres were assessed to achieve higher loading of diclosulam and good stability of microspheres.

Results: The mean diameter of xanthan gum-alginate microsphere was higher with 6 per cent ion gelation bath followed by the concentration of 4 and 2 per cent. Higher entrapment efficiency of diclosulam was achieved with loading of two percent diclosulam in six per cent calcium crosslinked microspheres

Conclusion: Xanthan gum- alginate microsphere system offers both burst release and controlled release of active ingredients. However, controlled release polymeric templates with herbicide will synchronize the release of herbicide with the emergence of weeds in the cropped situations for better weed management.

Keywords: Micro encapsulation, Xanthan gum, Sodium Alginate, Diclosulam, Ion gelation

1. INTRODUCTION

Herbicides are the second-largest category of plant protection chemical behind insecticides in terms of consumption. The Indian herbicide market had achieved the growth of 12.3 per cent since 2018, compared to the growth of 8.9 per cent for other insecticide and fungicide. The use of herbicides is the efficient strategy to manage weeds in crop production. However, the continuous and indiscriminate use of herbicides cause phytotoxicity in crops, faster

degradation due to light, persistence in the soil, and leaching into ground water. Overuse of herbicides affect succeeding crops through the carry over effect in the soil. The development of herbicide-resistant weed species, and shift in weed flora were observed due to the frequent use of herbicides to control weeds [1]. Up to 20 to 25 DAS, pre-emergence herbicides have proven to be very successful, but late-arriving weeds obstruct pegging, pod development, and harvesting [2]. The fate of herbicides in the soil determines the efficiency of herbicidal activity, where rapid conversion of intermediates in the soil making less effective against weeds. In addition, intermediate compounds from the degradation path of herbicides in soil contaminate the groundwater [3]. Diclosulam (2', 6'-dichloro-5-ethoxy7fluoro [1,2,4] triazolo[1,5c] pyrimidine2sulfonanilide) is a low volume herbicide belonging to sulfonanilide family. Diclosulam interfere with Aceto Hydroxy Acid Synthase (AHAS), a key enzyme involved in the synthesis of branched-chain amino acids such as leucine, isoleucine and valine in plants, thus affecting protein synthesis and cell division that leads to death of targeted weeds. Diclosulam is recommended as pre-emergence herbicide (soybean, groundnut, sorghum, potato, maize, wheat, barley and oats), applied at 1 to 3 days after sowing with the optimum dose of 20 g a.i. ha^{-1} for groundnut [2]. Phytotoxicity symptoms such as yellowing of leaves are reported in groundnut with the application of diclosulam. Similarly, pre-emergence application of diclosulam controls weeds up to 40 days of sowing in groundnut which warrants either hand weeding and post emergence application of herbicides to maintain weed free situation to prevent economic yield loss. Diclosulam binds with soil that leads to less mobility and leaching in soils causing less herbicidal activity. Hence, the research was proposed to design diclosulam systems for releasing active molecules at regulated manner to maintain the concentration of herbicide in soil to cause herbicidal action.

Encapsulation with natural polymers is the best strategy to improve weed control efficiency of herbicides and reduce herbicide adsorption in the soil. Microsphere encapsulation of agrochemicals in pectin based polymeric system was used for the smart release of herbicides [4]. Diverse properties of natural polymers were preferred for encapsulation of herbicides over synthetic materials due to non-toxicity in nature, abundance availability and the cost [5]. Xanthan gum and alginate are natural polymers, explored as a carrier for encapsulation of plant protection chemicals and microbes. Xanthan gum is a microbial extra cellular polysaccharide produced by Gram negative aerobic bacteria *Xanthomonas campestris* [6]. Xanthan gum is biocompatible in nature, bio adhesive, biodegradable , non-toxic and low cost which makes suitable candidate for the encapsulation of agrochemicals [7]. Biofilms, hydrogels, micro particles and nanoparticle of xanthan gum and xanthan gum based composites are widely used for designing controlled release systems [8]. Xanthan

gum acts as emulsifier and thickening agent besides its good water-control properties and pseudoplastic behavior, where xanthan gum at lower concentrations readily form high viscous solution exhibiting pseudoplasticity [9]. Sodium alginate is an anionic polysaccharide derived from sea algae (Phaeophyceae) that contain 30–60 per cent of alginic acid. Sodium salt of alginic acid is a linear polymer made up of residues of 1, 4-linked D-mannuronic acid and D-gluronic acid. Sodium alginate nanoparticles are also explored for designing pesticide delivery systems due to its biodegradability, biocompatibility, and low toxicity in nature. Therefore, the present study focuses on designing xanthan gum and sodium alginate based polymeric system for the delivery of diclosulam for improving weed control efficiency

2. MATERIAL AND METHODS

2.1. Materials

Sodium alginate (HiMedia: Cat No. RM7494) was purchased from HiMedia (Mumbai, India) while methanol (Merck: Cat. No. 494291) was obtained from Sigma Aldrich (Mumbai, India). IAMPURE Ingredients (Chennai, India) supplied xanthan gum. Calcium chloride anhydrous (Avra: Cat No. ASC2461)) was purchased from Avra Synthesis Private Limited. (Hyderabad, India), Phosphate buffer was procured from Molychem (Mumbai, India), Diclosulam herbicide (84% WDG) and ethanol were purchased locally. None of the chemicals was further purified before being employed in the study.

2.2. Methods

2.2.1. Encapsulation of diclosulam in Xanthan gum - Alginate microspheres

Xanthan gum (XG) – Sodium alginate (SA) microspheres were prepared based on the earlier protocol [10] with some modifications through ionotropic gelation method. A homogeneous solution of xanthan gum and sodium alginate were prepared by dissolving in water using a reflux condenser at 45°C. The ratio of sodium alginate to xanthan gum was kept as 1:0.67 for the preparation of microspheres. The XG/SA mixture was dropped to ion gelation bath of calcium chloride (2, 4and 6 per cent) to obtain microspheres. The distance between the delivery point of syringe and ionic solution was maintained at 7cm, while flow rate of polymer mixture was kept at 16 drops minute $^{-1}$. Microspheres were cured in the gelation bath for 45 minutes in the room temperature. Subsequently, microspheres were washed with deionized water to remove non-cross-linked cations over the surface of the beads. Beads were dried at room temperature for 48h until obtaining uniform weight of beads. Diclosulam was homogenized in the polymer matrix before dropping into gelation bath to facilitate the encapsulation of diclosulam in the polymeric systems

2.2.2. Entrapment efficiency of Diclosulam in Microspheres

Encapsulation efficiency represents the amount of active ingredient encapsulated in matrix complexes of xanthan gum-alginate polymeric systems. Herbicide loaded beads of varying concentrations were analyzed individually using UV-Vis spectrophotometer (Analytik Jena, Specord 210 Plus). Diclosulam (84 per cent WDG) was prepared to 10 ppm using 0.067 M phosphate buffer (6.8 pH) for assessing the wavelength of maximum absorption through scanning with a wavelength of 200 to 800 nm in UV Vis spectroscopy. The known concentrations of diclosulam (5, 10, 15, 20, 25 and 30 ppm) standards were used to find OD values in UV Vis spectroscopy for obtaining standard curve. Similarly, microspheres (20 mg) containing diclosulam were dissolved in 10 ml of 0.067 M phosphate buffer (6.8 pH) for 4h for complete dissolution of microspheres. Aliquot was filtered through membrane filter (0.22µm) to reduce the residual interaction and analyzed at λmax = 251 nm using a spectrophotometer. Diclosulam entrapment efficiency was calculated using the following formula

$$\text{Encapsulation Efficiency (\%)} = \frac{\text{Actual herbicide content}}{\text{Theoretical herbicide content}} \times 100$$

2.2.3. Swelling behavior and water uptake of microspheres

The swelling property and water uptake of diclosulam-loaded xanthan gum – alginate microsphres were assessed in terms of change in mass and diameter with function of time. Measurements of the initial diameter and mass of dried microspheres were made before beads were soaked for two hours at room temperature in phosphate buffer (6.8 pH). The microspheres were retrieved from phosphate buffer in a regular intervals to assess mass and diameter at time‘t’. The percent of water uptake and swelling per cent were calculated using following equations

$$\text{Percent Swelling} = \frac{\text{Diameter of microsphere at time "t" – Initial diameter of microsphere}}{\text{Initial diameter of microsphere}} \text{ X } 100$$

$$\text{Water uptake} = \frac{\text{Weight of microsphere at time " t " - Initial weight of microsphere}}{\text{Initial weight of microsphere}} \text{ X } 100$$

2.2.4. Characterization of diclosulam loaded xanthan gum -alginate microspheres

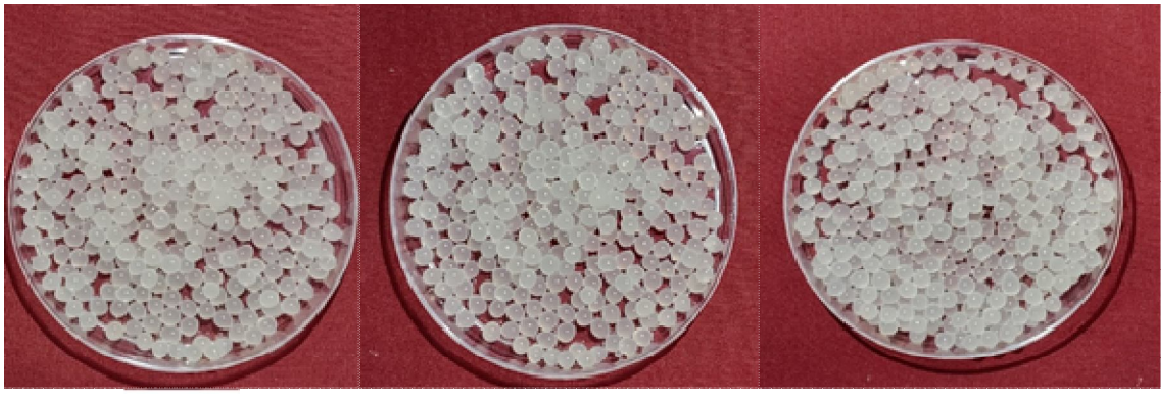
Diclosulam loaded xanthan gum – alginate microspheres were examined under digital optical fluorescent microscope (ProgRes C5 indi) at 5x magnification to determine the size of microsphere. The surface topography of microspheres was assessed using scanning electron microscope (Quanta 250, FEI, Czech Republic). Electron microscopy images were obtained by mounting the microsphere on carbon stub at an acceleration voltage of 10kV with chamber pressure of 1.0mm Hg. Fourier Transformed Infra-Red spectra of xanthan gum, sodium alginate, diclosulam, xanthan gum – alginate microsphere and diclosulam loaded xanthan gum – alginate microsphere was recorded using JASCO –FTIR-6800.

Attenuated total reflection (ATR) mode was used at wavenumber regime of 4000 to 400 cm^{-1} with 4 cm^{-1} resolution and 32 scans. The pore size, pore radius and surface area of microspheres were assessed using Brunner –Emmet-Teller (Quantachrome NOVAtouch NT2LX-1). The microspheres were degassed at 100°C for 3h to remove moisture and subjected to the adsorption of nitrogen gas at serious of pressure points (0 to 800 torr).

3. RESULTS AND DISCUSSION

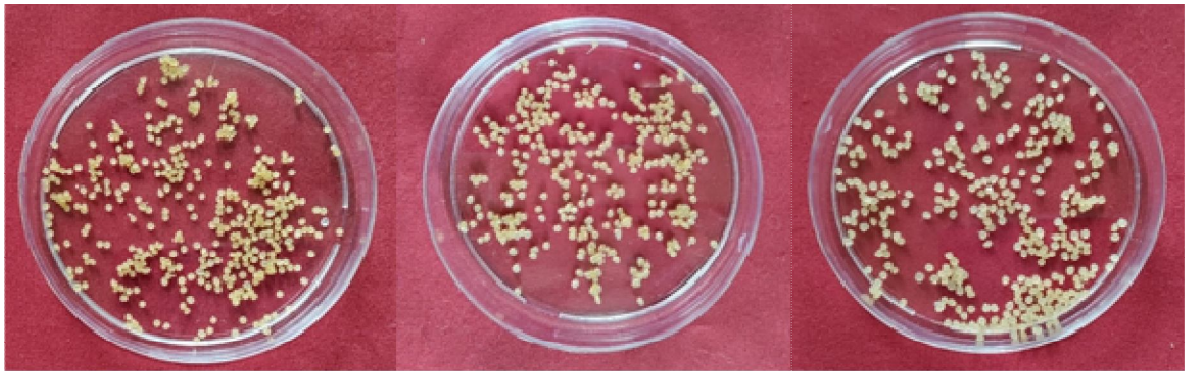
Encapsulation of diclosulam in xanthan gum –sodium alginate polymeric microcapsules or microspheres was carried out through ionic gelation technique. The concentration of ion gelation bath affected the size and behaviour of xanthan gum –sodium alginate microspheres. Fresh and dried xanthan gum - sodium alginate microsphere without loading diclosulam cross-linked with various concentration of ion gelation bath (calcium chloride) are illustrated in Fig 1a, 1b, 1c and 2a, 2b and 2c. The microspheres were completely spherical in nature irrespective of ion gelation bath concentrations. Gelation occurs through ionic bonding between divalent cations in the gelation bath and carboxyl group of guluronic acids in sodium alginate and mannose units in xanthan gum. Gelation process results in the formation of three-dimensional structures, which further are stabilized through the exchange of sodium ions in the polymer matrix with divalent calcium ions in the gelation bath. The rate of divalent cations in gelation bath determines the spherocity of microspheres. The regain of spherical shape of polymer beads in the ion gelation bath occurs when the viscous force of polymers overcome the drag force of ion gelation bath [10]. Hence, the concentration of xanthan gum and sodium alginate used in the study creates viscous force to regain the spherical shape of beads through overcoming drag forces of ion gelation bath. Chloride ions interact with uronic acid of sodium alginate to form calcium alginate xanthan layer externally over the bead exchanging sodium ions. The digital fluorescence microscopic image of calcium chloride cross-linked xanthan gum-alginate microspheres shows a uniform undulation (black color) from periphery to center of the bead, which was cross-linked with two per cent of ion gelation bath (fig.3a). On the other hand, thick outer shell and smooth inner core were observed in beads cross-linked with, six per cent ion gelation bath (fig.3c). The wall materials surrounding the core molecules (herbicide) attribute to the greater improvement of physical and chemical stability and control the delivery of core materials [11, 12]. Matrix polymeric type delivery system (microspheres) offers the controlled release of core material through equidistant matrix erosion after a burst release at initial phase of delivery. Hence, the effects of calcium chloride concentrations (ionic cross linker) were observed in the digital images of fluorescent microscope. Higher rate of divalent ions complexes readily with peripheral layer of polymer matrix, thus resisting further penetration of

divalent calcium into center of beads resulting in poor crosslinking. Lower concentration of divalent ions in the gelation bath allows the movement of cations into beads completely to achieve higher crosslinking. The intensity of black coloration in the images indicates the intensity of calcium ions, where beads cross-linked with six per cent calcium chloride results in the thick black peripheral layer over the bead representing higher concentration of calcium ions and vice versa.



[1a] [1b] [1c]

Fig 1: Fresh Xanthan gum alginate microspheres: [1a]: 2% calcium chloride cross linked; [1b]: 4% calcium chloride cross linked; [1c]: 6% calcium chloride cross linked



[2a] [2b] [2c]

Fig 2: Dried Xanthan gum alginate microspheres: [2a]: 2% calcium chloride cross linked; [2b]: 4% calcium chloride cross linked; [2c]: 6% calcium chloride cross linked.

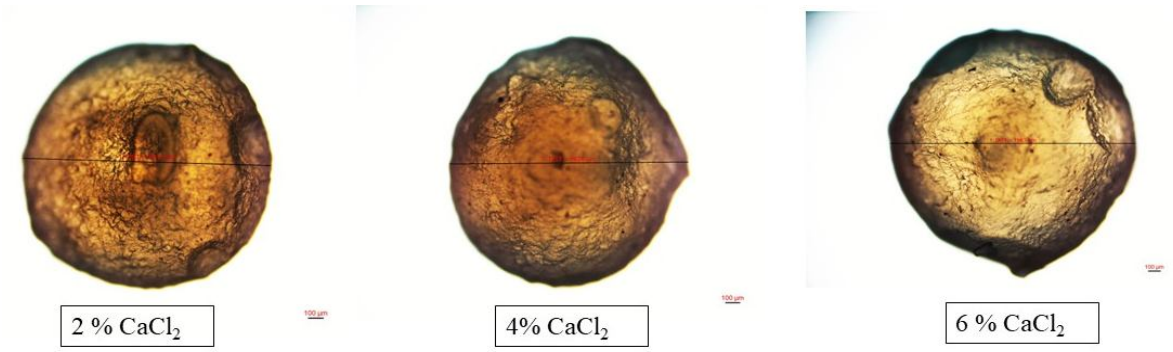
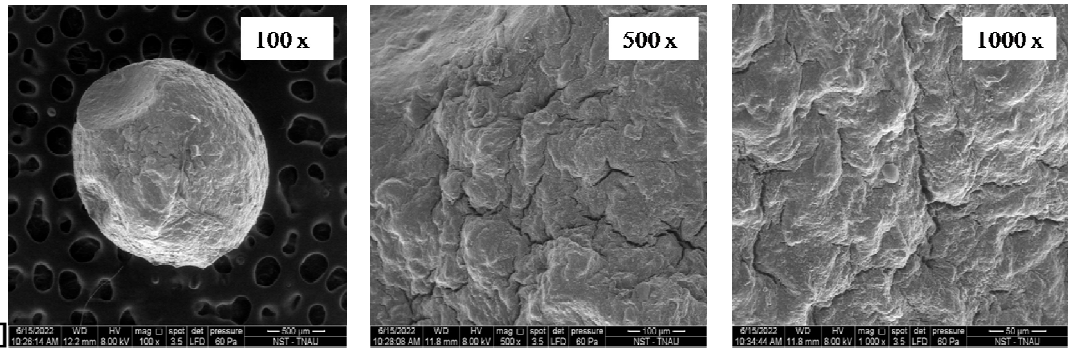


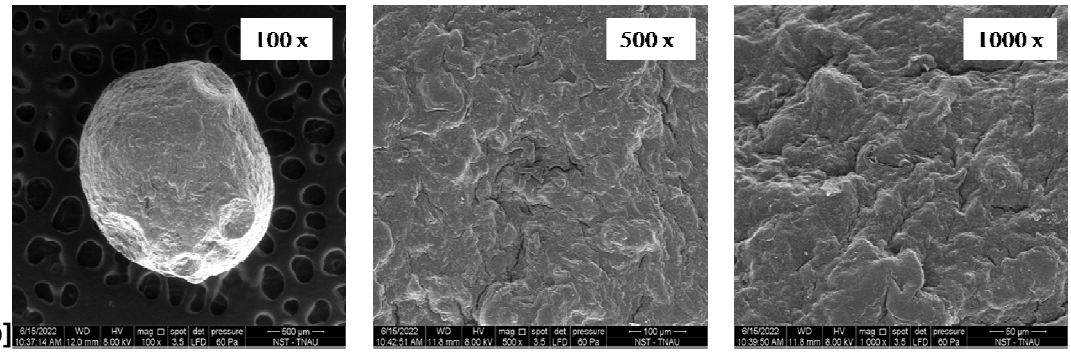
Fig 3: Digital optical fluorescent microscopic image of two, four and six percent Calcium chloride cross linked Xanthan gum – Alginate microsphere

A uniform influx of calcium ions with efflux of water and sodium ions occurs at low concentration of ion gelation bath, whereas maximum cross-linking forms with higher concentration of calcium chloride at outer layer of polymer matrix (bead) that becomes hardened and prevents the further influx of calcium ions for cross linking, thus forming a smooth inner core.

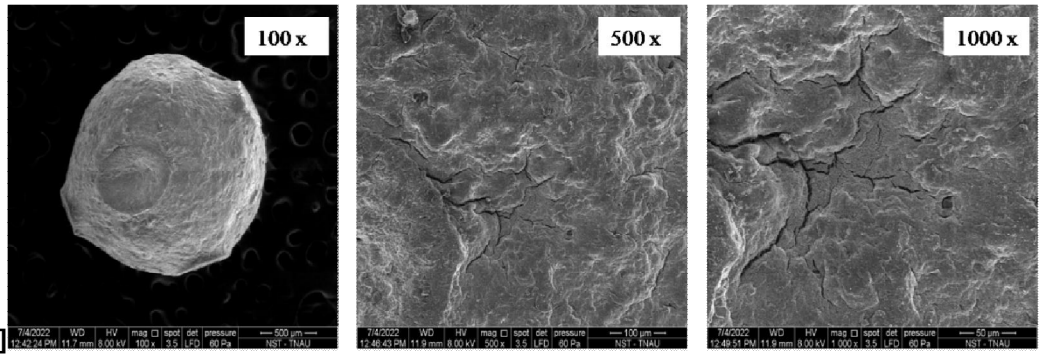
Scanning electron microscopic images of xanthan gum sodium alginate microspheres are illustrated in fig.4., which substantiate the above statement with a gradual decline in cracks or pinholes with increasing concentration of calcium ions in ion gelation bath. The shape and average diameter of xanthan gum-alginate microspheres are tabulated (Table1), which indicates the mean diameter of microsphere increases with increasing concentration of calcium chloride. Calcium ion influx, force efflux of water molecules and makes the microspheres to downsize resulting in smaller bead size at lower concentration of ion gelation bath. The mean diameter of beads, which were cross-linked, with concentration of six per cent calcium chloride resulted in larger bead size (2.00 mm) compared to beads (1.61 mm) cross-linked with two per cent concentration of calcium chloride.



[4a]



[4b]



[4c]

Fig 4: SEM micrographs of Calcium chloride cross linked Xanthan gum-alginate microsphere; [a]: 2% $CaCl_2$ Cross linked; [b]: 4% $CaCl_2$ Cross linked; [c]: 6% $CaCl_2$ Cross linked.

Table 1: Effect of various concentration of calcium chloride on mean diameter and shape of xanthan gum alginate microspheres

Concentration of Calcium chloride (%)	Diameter (mm)	Remarks
2 per cent	1.61	Beads were round and spherical
4 per cent	1.63	Beads were round and spherical
6 percent	2.00	Beads were oval

Pore volume and surface area governs the water uptake and dissolution of active molecules loaded in the polymeric matrix system. Cross linking of polymers with different concentration of ionic solution influence the surface area and pore volume of microspheres (Table 2). The surface area (1.92 m^2 g^{-1}) and pore radius (2.94 nm) of microspheres were higher with crosslinking of two per cent calcium chloride, while beads complexed with six per cent calcium chloride have less surface area (1.05 m^2 g^{-1}) and pore radius (2.44 nm)

Thus, ionic solution concentration is inversely proportional to pore volume and surface area. This implies that, a greater number of functional groups in polymers reacted at increasing concentration of ionic solution resulting in increased bead size. Hence, downsizing of beads with lower concentration of cross linkers proved with higher surface area, while uniform

crosslinking throughout the polymer matrix in lower concentration of calcium chloride promotes higher pore radius. The type I isotherm (fig.5) of calcium chloride cross-linked beads exhibits monolayer adsorption of gaseous molecules confirming the micro porous nature of polymeric beads. Hence, the pore volume and surface area are foremost parameter determining the release of core materials into the medium.

Table 2: Effect of various concentration of calcium chloride on pore volume, pore radius and surface area of xanthan gum alginate microsphere.

Treatments	Pore volume (cc/g)	Pore radius (nm)	Surface area (m^2/g)
Calcium chloride 2%	0.024	2.94	1.92
Calcium chloride 4%	0.017	2.94	1.45
Calcium chloride 6%	0.011	2.44	1.05

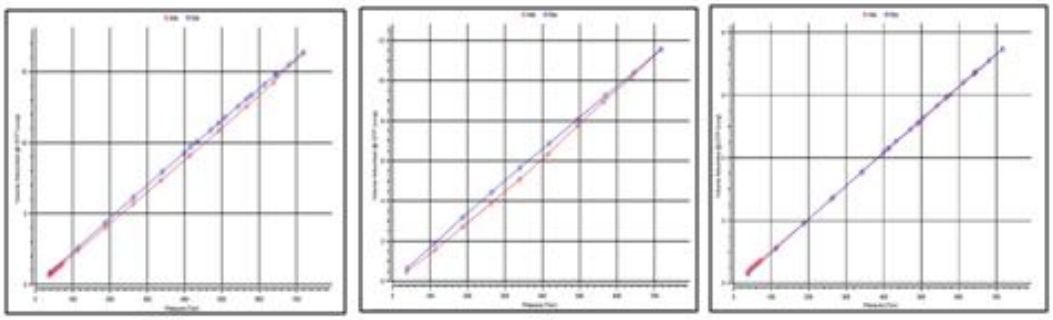


Fig 5: Effect of Calcium chloride concentration on absolute isotherms of xanthan gum-alginate microsphere.

Further, diclosulam (hydrophobic moiety) was encapsulated in xanthan gum alginate microspheres. The gelation of polymeric molecules entraps diclosulam simultaneously through gelation with calcium divalent ions. Thus, ionic bond formed between calcium ions and, carboxyl and hydroxyl groups of polymers favoring the formation of “egg box” complex [13-16]. The three-dimensional structures get stabilized through exchanging sodium ions with calcium ions in the polymeric system, provided maximum stability by minimizing internal entropy[17], represented in the infra-red spectra of diclosulam loaded polymeric system (fig 6) forming strong bridging complex with disappearance of 1407cm^{-1}peak corresponding to OH bending of carboxylic acid.

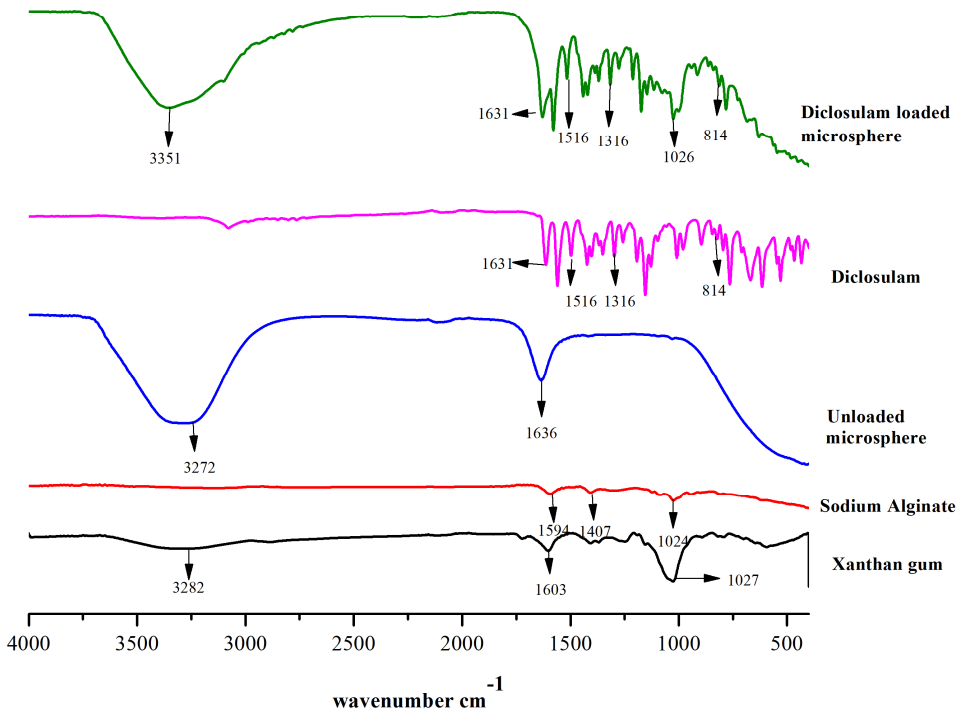
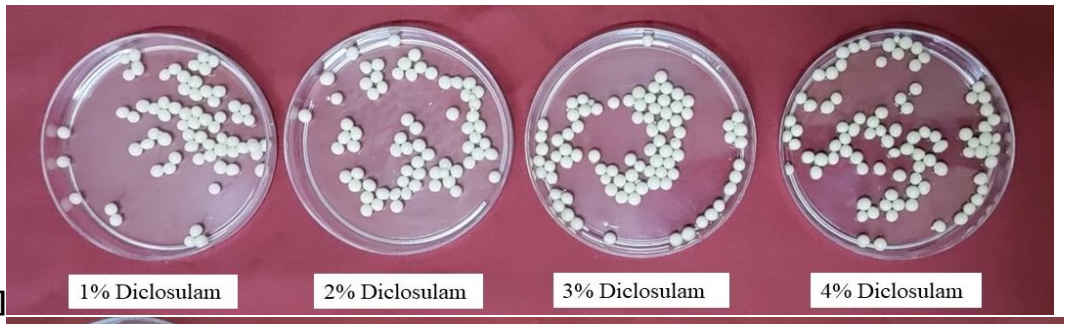


Fig 6: FTIR spectrum of xanthan gum, sodium alginate, xanthan gum – alginate microsphere, diclosulam and diclosulam loaded xanthan gum – alginate microsphere

The calcium ions offer instantaneous formation of bridging complex between carboxyl groups of xanthan gum – sodium alginate polymeric system, indicates the formation of partial covalent bond with decrease or disappearance of COO^- stretching peak at 1024 cm^{-1} of sodium alginate and shift to higher wavenumber 1631 cm^{-1} confirming the complexation process in cross-linked polymeric system [18-20]. The intermolecular hydrogen bonding formed between sodium alginate and xanthan gum was more prominent with an increased intensity peak at 3272 cm^{-1} in the cross-linked polymeric system [21, 22]. The physical adsorption of diclosulam to xanthan gum-alginate microspheres were confirmed through the presence of characteristic vibrational peaks of 1578, 1516,850 to 550 corresponding to crystalline nature and C-Cl stretching of halo compounds in diclosulam loaded polymeric system (Fig 6). Different concentrations of diclosulam were loaded in xanthan gum alginate microspheres. The fresh and dry beads of diclosulam loaded xanthan gum alginate microspheres were shown in fig 7&8 respectively. The mean diameter of diclosulam loaded xanthan gum-alginate microsphere increases with increasing concentration of diclosulam (Table 3). Bead diameters of diclosulam-entrapped microspheres were increased with

concentration of diclosulam up to three per cent, whereas microspheres with four per cent of diclosulam resulted in lower size irrespective of concentration of ion gelation bath. Mean size of microspheres with one percent diclosulam was 2.12 mm, whereas the average diameter of beads with three per cent diclosulam was 2.30 mm. The size of microspheres with loading of diclosulam with four percent concentration was 2.10 mm, indicating the lesser size of beads with higher concentration of diclosulam beyond three per cent. Mass of diclosulam loaded microspheres increases gradually with increasing concentration of diclosulam. However, the discrepancy in mass and diameter of microspheres loaded with four per cent diclosulam might be due to the increased specific gravity of polymer solution.

[7a]



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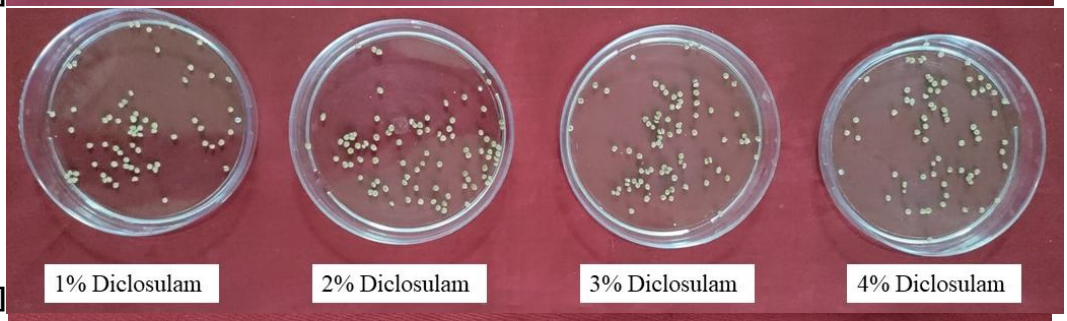

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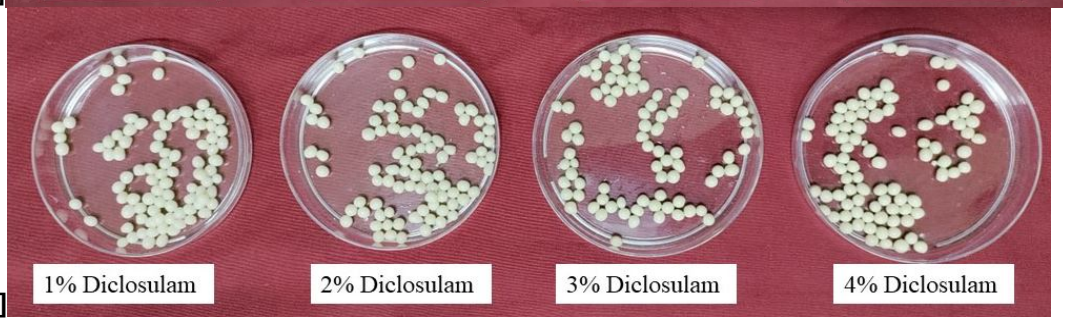

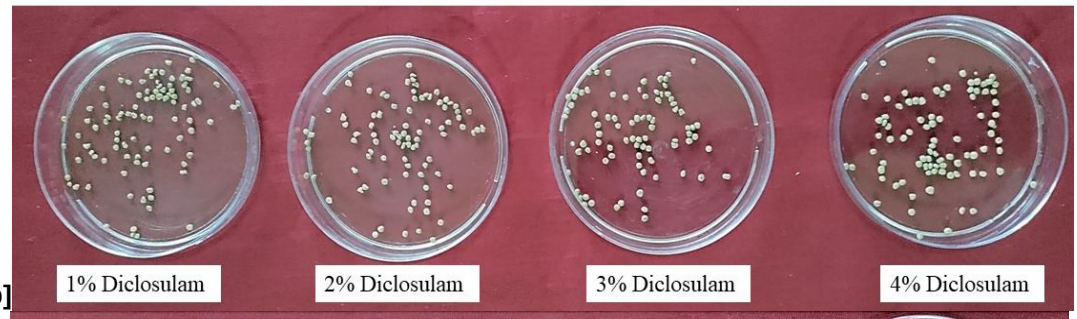

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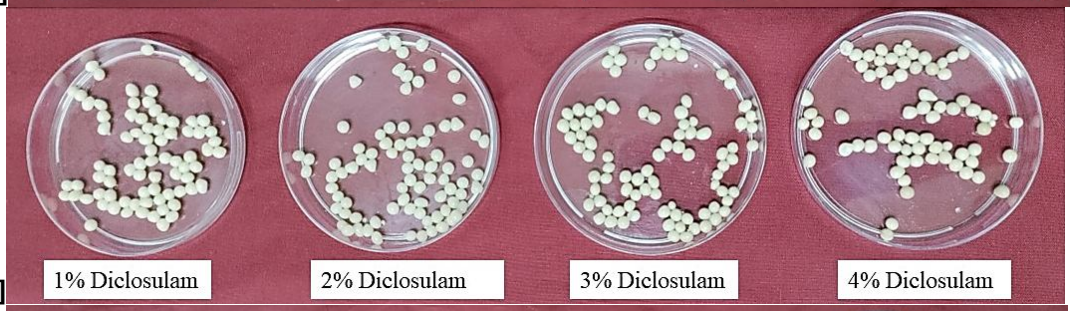

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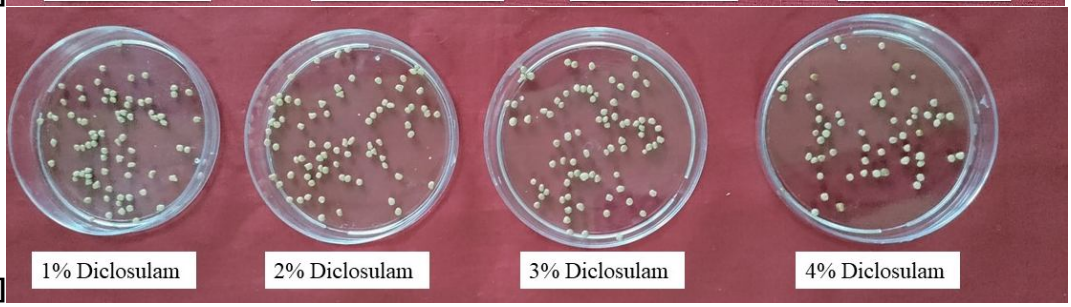


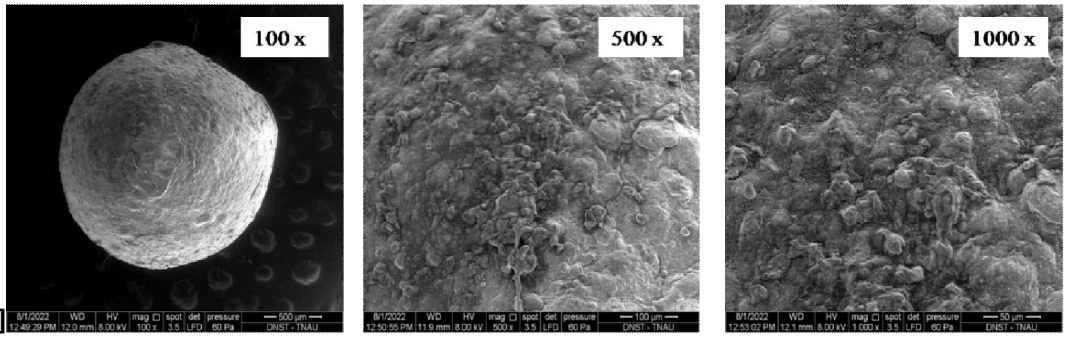
[8c]

Fig 7&8: Diclosulam loaded Xanthan gum-Alginate fresh and dry microsphere respectively; [7a&8a]: 2% $CaCl_2$ crosslinked; [7b&8b]: 4% $CaCl_2$ Crosslinked; [7c&8c]: 6% $CaCl_2$ crosslinked

Table 3: Effect of calcium chloride and diclosulam concentrations on mean size and shape of xanthan gum alginate microspheres

Calcium chloride	Diclosulam	Diameter (mm)	Remarks
2 percent	1 percent	1.99	Beads were round and spherical
	2 percent	2.16	Beads were round and spherical
	3 percent	2.26	Beads were round
	4 percent	2.02	Initially beads were round and turns oval upon increasing the gelation time
4 percent	1 percent	2.14	Beads were round and spherical
	2 percent	2.26	Beads were round and spherical
	3 percent	2.28	Beads were round
	4 percent	2.16	Initially beads were round and turns oval upon increasing the gelation time

6 percent	1 percent	2.25	Beads were round and spherical
	2 percent	2.27	Beads were round and spherical
	3 percent	2.38	Beads were round
	4 percent	2.13	Initially beads were round and turns oval upon increasing the gelation time



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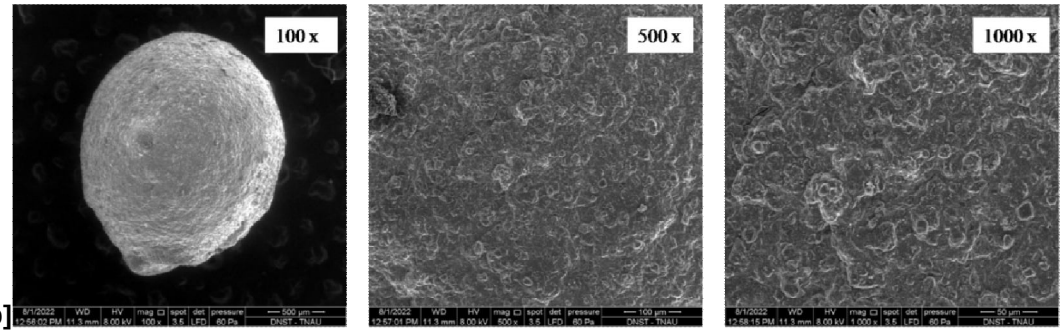

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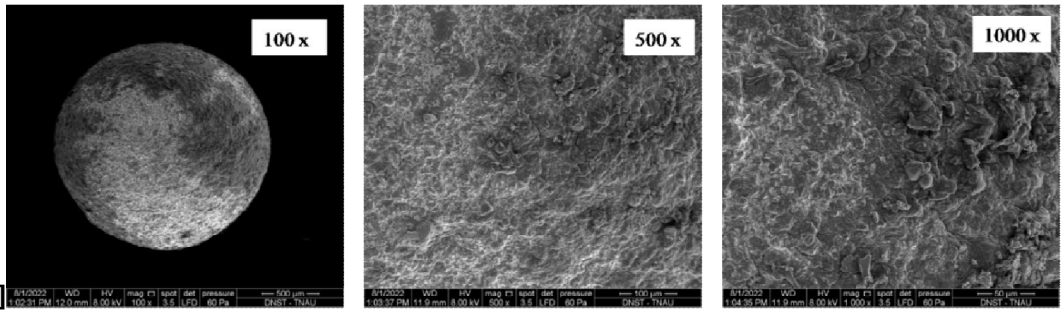

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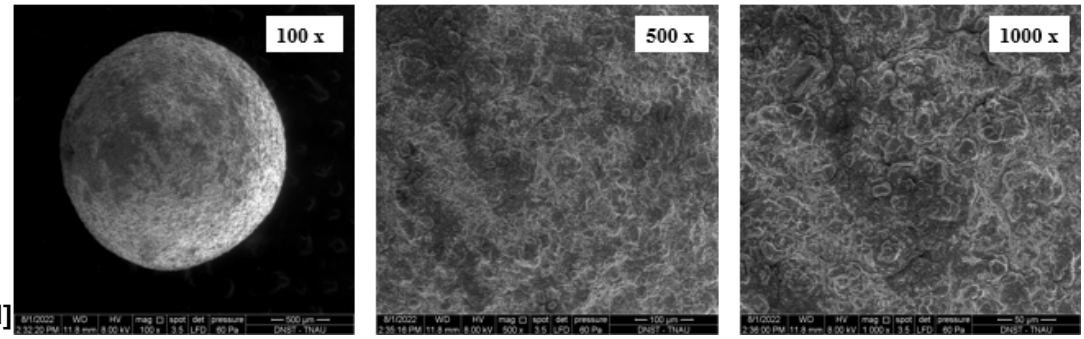


[9d]

Fig 9: SEM micrographs of diclosulam loaded in two percent calcium chloride crosslinked xanthan gum alginate microsphere; [a] 1% Diclosulam loaded; [b]: 2% Diclosulam loaded; [c]: 3% diclosulam loaded; [d]: 4% diclosulam loaded.

Scanning electron microscopic images of diclosulam loaded polymeric system revealed that, appearance of fringes of xanthan gum – alginate microspheres and exhibition of protrusions over the surface of microspheres forming an undulated surface which signifies the presence of particulate matter. The intensity of particulate matter increases with increasing concentration of diclosulam (fig 9) loaded in the polymeric system. A comparison of entrapment efficiency of various concentration of diclosulam loaded in xanthan gum – alginate microsphere was shown in fig 10. The entrapment efficiency of diclosulam decreases with increasing concentration of diclosulam in xanthan gum alginate microspheres. The higher concentration of diclosulam increases the polymer to drug ratio, thereby it reaches saturation and declines the entrapment efficiency. Higher entrapment efficiency (69.6 and 70.3 per cent) of diclosulam was noticed in six percent and four percent calcium cross linked microspheres loaded with two percent diclosulam followed by two per cent calcium cross linked microspheres with one per cent diclosulam with an entrapment efficiency of 58.6 per cent. The lowest entrapment efficiency was observed in microspheres with loading of diclosulam @ four per cent, might be due to efflux of diclosulam molecules out of polymeric system to achieve equilibrium diffusion coefficient with surrounding ion gelation bath. Conversely, [23, 24] reported that, entrapment efficiency decreases with increasing concentration of crosslinkers.

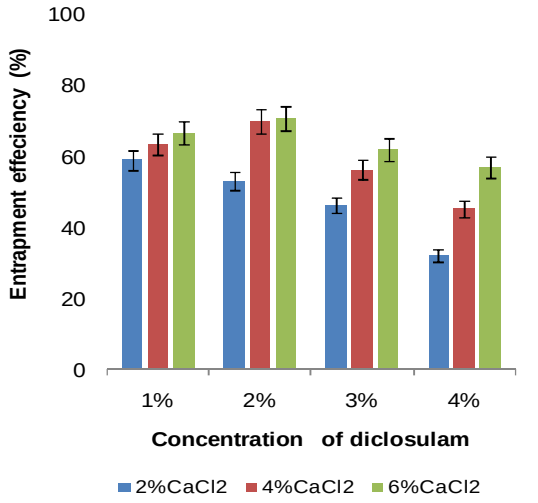
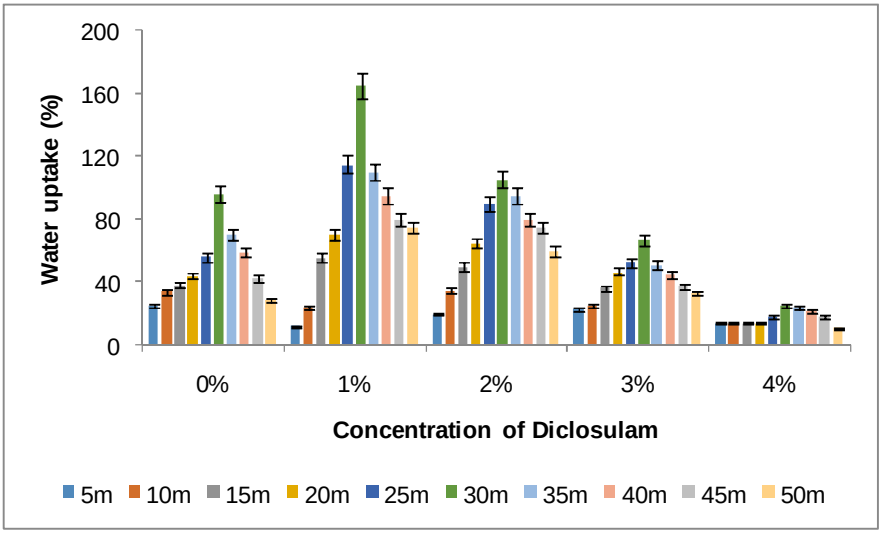


Fig 10: Effect of calcium chloride and diclosulam concentration on entrapment efficiency of diclosulam in Xanthan gum alginate microsphere

Water uptake in the polymeric systems determines release kinetics of entrapped functional materials. The effect of various concentrations of calcium chloride on water uptake of diclosulam-loaded microspheres was shown in fig.11. The percent of water uptake increases with time in microspheres with irrespective cross linker concentration. The higher water uptake was observed at 35th minute in microspheres cross-linked with two per cent concentration of calcium chloride followed by four per cent. However, microspheres formed with six per cent calcium chloride get dissipated in water medium within 10 minutes of incubation.



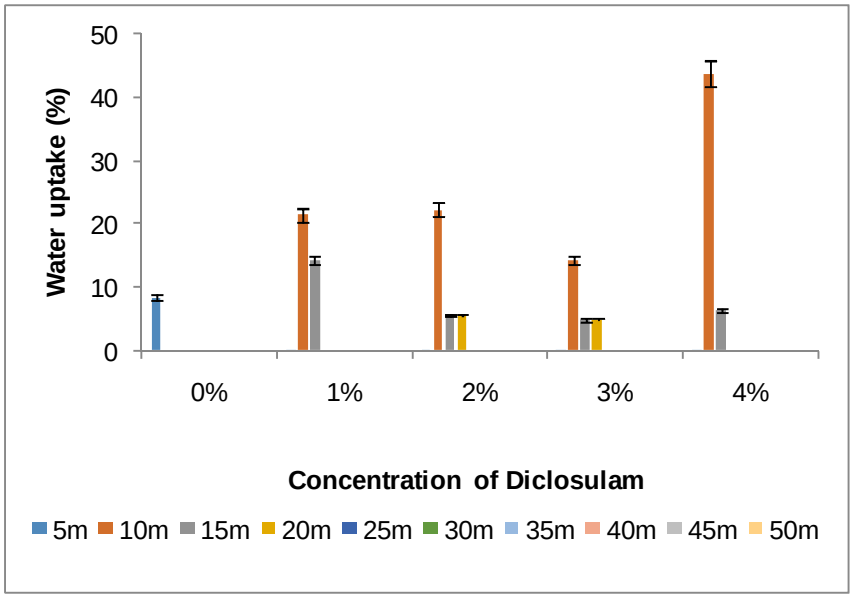


Fig 11: Effect of different concentration of calcium chloride on water uptake of diclosulam loaded (1%, 2%, 3% & 4%) and unloaded xanthan gum-alginate microsphere; [a]: 2% $CaCl_2$ crosslinked; [b]:4% $CaCl_2$ crosslinked; [c]: 6% $CaCl_2$ crosslinked;

Lesser water uptake in microspheres cross-linked with six per cent of calcium chloride was due to high degree of cross-linking over the surface. Swelling studies of microspheres indicated that beads cross-linked with lesser concentration of calcium chloride (2 and 4 per cent) absorbs water and reached maximum uptake of water during 30 to 35 minutes of incubation, while microspheres with six per cent attained maximum water uptake with 10 minutes of incubation. Slower water influx was observed in the polymeric microsphere matrix formed with lesser concentration of calcium during the initial phase of incubation and reaches saturation with completely disintegrated microsphere polymer. Microspheres cross linked with higher concentration of calcium attain saturation rapidly and dissolves with burst release of functional molecule through poorly crosslinked core region[25].

4. CONCLUSION

This study revealed that calcium mediated ionotropic gelation of xanthan gum-alginate composite resulted in spherical and stable beads possessing ability for the sustained release of diclosulam. The mean diameter of xanthan gum microsphere increased from 1.61mm to 2.00 mm with increasing concentration of calcium ions in gelation bath. The entrapment efficiency of diclosulam @ two percent was higher with crosslinking of 4 and 6 per cent calcium gelation bath, however, microspheres cross-linked with higher concentration achieved burst release of diclosulam due to complete dissolution in 10 minutes of incubation

in water medium. The microspheres with lower cross linker concentration offers controlled release of active molecules, which proved from the water uptake studies. Burst releases of mechanism of microspheres are not for the delivery of diclosulam herbicide due to localized release of diclosulam from microspheres that induces phytotoxicity. Microspheres cross-linked with two and four per cent calcium chloride offers controlled release of diclosulam, which synchronize with crop growth duration, will not cause phytotoxicity in crops and achieve prolonged weed control.

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COMPETING INTERESTS

Authors have declared that no competing interest exists.

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