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Development and evaluation of Agri-glasses as slow release fertilizers

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Abstract
A number of Agri-glass compositions were designed and formulated along their related scientific investigations in the laboratory. The prepared glass compositions were divided as solid samples for different investigations and remaining were powdered after a ball milling for 30 minutes (approximately) for the agriculture applications. Rate of leaching of various constituents present in the prepared glass composition had also been carried out immersion test method. FTIR and SEM techniques have also been employed to explain the data obtained.

Keywords: Glass fertilizers, controlled release, macro and micro glass nutrients, hydrolysis

Introduction
Vitreous fertilizers are advanced and having a tendency of controlled released of a number of elements like - K, P, Mg, S, Ca along with a micronutrient like B, Fe, Mo, Cu, Zn, Mn, V, which are essential not only for growth of the plant but also helpful to complete their physiological functioning and addition in the quantity of micronutrients addition in the glass networks in their oxide form and other required chemical forms ranges from 5 to 10 moles per cent as per agriculture requirement (Chaturvedi *et al., 2014,*Takker *et al*, 1978) [1, 2]. The role of different plant nutrients is very specific considering the aspect in terms of agriculture (Waclawska and Szumera, 2009; Torrisi *et al.,* 2013; Chaturvedi *et al.,* 2008; Javed *et al.,* 2013) [3, 4, 5, 6].

The objective of the research is to minimize the use of various types of chemical fertilizers applications and provide a parallel environmental friendly fertilizer through glass matrix with enhancing the agriculture productivity as usual without leaving any kind of toxicity and cost effective also (Stoch *et al*., 2003, and Trenkel, 2010) [7, 8].

Experimental
Analytical and reagent grade chemicals as raw materials were used for the preparation of glass compositions. Batches of glass compositions were melted in alumina crucibles of 100 ml capacity in an electric furnace at 900℃±5℃. The melts were then poured on aluminum plate (as base) within a metal made mould. After getting it dried, stored in a desiccator. The half portion of glass prepared was separated for getting powder with the help of planetary ball mill upto micro to nano-size particles for agriculture field applications. After ball milling the powder passed through sieves of 45 µm particle size. Another half portion of prepared glass composition in the form of solid samples were utilized for leaching experiments. These samples were hanging with help of a thread to get maximum exposure of all the surfaces of the samples which provide an easy passage for release the species/constituents present in the glass network. After immersion in natural water for different period of time in a digital hot air chamber maintaining the temperature at 35℃ throughout the period of investigations. Glass specimens were also investigated for the presence of nature of bonds formations in the glass compositions and after the leaching of various species and constituent from the glass network or matrix in the form of deficiencies by using the technique FTIR. The surface morphology of all prepared glass composition was also carried out before and after leaching experiment with the help of SEM.

Results and Discussions

During the required duration of time weight losses were measured after 15, 30, 45, 60 days immersion of glass samples into water system and their related observations are summarized in the table 3.

Table 1: Compositions of the glass samples in mole%

Sample	P2O5	SiO2	B2O3	K2O	CaO	MgO	FeO	ZnO	CuO	MnO
NG1	35	10	10	25	10	10	-	-	-	-
NG2	35	10	10	25	5	5	10	-	-	-
NG3	35	10	10	25	5	5	-	10	-	-
NG4	35	10	10	25	5	5	-	-	-	10
NG5	35	10	10	25	5	5	-	-	10	-
NG6	35	10	10	15	5	5	5	5	5	5

Table 2: Compositions of the glass samples in weight%

Sample	P2O5	SiO2	B2O3	K2O	CaO	MgO	FeO	ZnO	CuO	MnO
NG1	51.88	6.26	7.31	24.53	5.85	4.18	-	-	-	-
NG2	50.61.06	6.11	7.13	23.93	2.85	2.04	7.33	-	-	-
NG3	50.15	6.05	7.07	23.71	2.83	2.02	-	8.17	-	-
NG4	50.66	6.12	7.14	23.96	2.85	2.04	-	-	-	7.24
NG5	50.22	6.06	7.07	23.74	2.83	2.02	-	-	8.08	-
NG6	51.34	6.2	7.23	14.57	2.89	2.07	3.72	4.18	4.13	3.67

The weight of the samples before and after leaching can help to find out the absolute weight loss of the glass samples and the rate of leaching per day due to loss of various species/ constituents from glass network. Naturally, this kind of leaching behavior investigated at laboratory level having a direct scientific significance with the agriculture scientific parameters observed.

It is evident from the table 3 that leaching aspects related to various constituents/ species present in the formulated glass network is taking place by the penetration water molecules for different duration of time his is indeed a good resemblance with motives of research. It is also clear here that in the beginning the leaching rate is high which gradually becomes slower in later course of investigation and finally, better rate of leaching was observed for NG4 consistently. This may be due to the lowest density which indicates about weak bond formation of glass network in this particular composition.

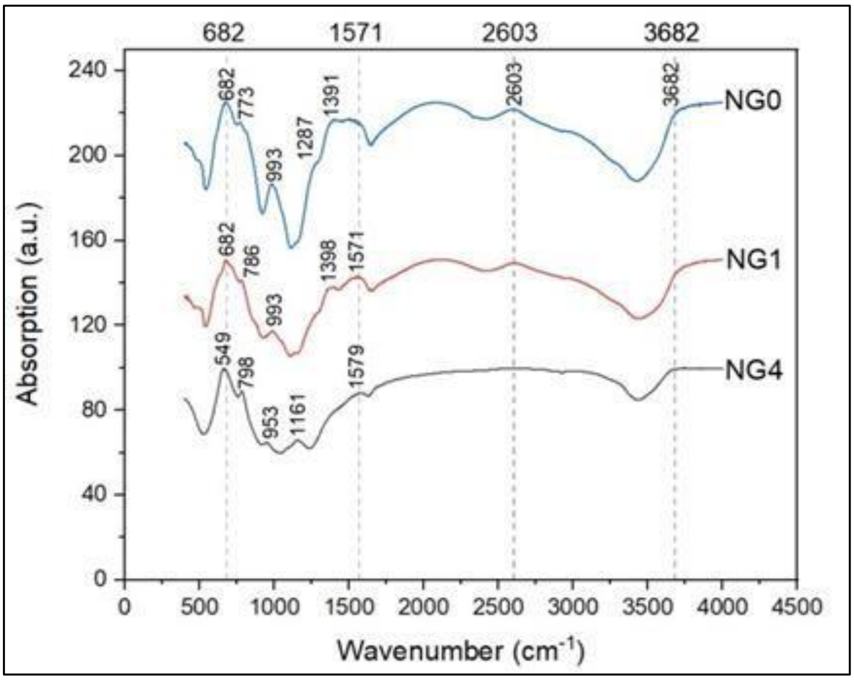

Fig 1: FTIR Spectra for NG0, NG1, NG4

The FTIR result of the glass nutrients clearly supports the leaching process as there is change in the absorption spectra of different bonds formed before and after leaching process. The FTIR results also show the possible bonds in the matrix. Siloxanes show one or more very strong infrared bands in the region of 900-1130 cm^{-1}. Disiloxanes and small-ring cyclo siloxanes show a single Si— O—Si band. As the siloxane chains become longer or branched, the Si—O—Si absorption becomes broader and more complex, showing two or more overlapping bands. The wavenumber between 860-940 cm^{-1} denotes the symmetric stretching vibration of P-O-P linkage. The region 1235–1293 cm^{-1} is a characteristic feature for the asymmetric stretching vibrations of the P=O bond. The region in 600-1000 cm^{-1} represents the B-O-B linkage in the matrix.

While the region between 950-1165 cm^{-1} represents stretching vibration of B-O-Si linkage.

After overall study of the FTIR analysis plots, it can be observed that the bond formations are initially taking place during the glass network formations which are losing their intensity after leaching because of the mobility of some species from glass networks during their exposure in water system. While comparing the FTIR analysis plots, particularly in the wave number range from 900-1300 cm^{-1}, 2500-2650 cm^{-1} and 3500-3700 cm^{-1} the specimens investigated for glass compositions NG0, NG1and NG4, there is a disappearance of peaks or being shorter in their size is clearly indicated that there is a loss of some species/constituents from glass network along with weaken of bonds present in the network after the penetration of water molecules during their exposure time.

These observations have a good resemblance with the results obtained by other processes carried out of the study.

In aqueous media the dissolution may takes place by the following reaction due to breaking of silicon–oxygen network (Bansal and Doremus, 1986) [9].

H2O + -Si-O- -------> -SiOH.HOSi-

Similar investigation have reported by other researchers (Cox and Khooli, 1992; Cox and Ford, 1989) [10, 11] when glass specimens were exposed in soil and water for a long duration of time initially, dissolution of glass will start (via ion exchange) of some weak bonded species from the glass network (RK Chaturvedi *et al*, 2021) [12]. Further, the monovalent cations present in network are more easily diffused because of their mobility in comparison doubly charged (divalent) and triply charged (trivalent cations) which are tightly bound with the glass network. In the similar way divalent and trivalent cations may migrate out from the glass network and finally tetravalent will be leached out with ultimate destruction of network (Chaturvedi *et al*., 2020) [13]. Considering the surface morphology among the micrographs before and after leaching for exposure to water system for 60days, it is clear from the comparison of the micrographs that some species have migrated out from the glass network in the case of both samples of vitreous fertilizers. (Burnie *et al*., 1981; Burnie *et al*., 1983 and Pyare, 2003) [14, 15, 16] In addition to this, it is also evident while going through the micrographs that the greater destruction in the surface morphology was observed in case of NG4 which has an almost good resemblance with the results obtained for rate of leaching research. It is also evident from the figure 3.

The objective of the research is to minimize the use of various types of chemical fertilizers applications and provide a parallel environmental friendly fertilizer through glass metric with enhancing the agriculture productivity as usual without leaving any kind of toxicity and cost effective also.

Table 3: Absolute weight loss and rate of leaching for different duration of time

Sample No. & Density	Immersion Time (days)	Initial Wt. (gm)	Final Wt. (gm)	Wt. loss (gm)	Rate of leaching (gm/day)	Remarks
NG1 Density-2.35	15	0.877	0.845	0.032	2.13 x 10-3	Breaks into small pieces along with white ppt. in second interval of immersion
	30	0.845	0.65	0.195	1.3 x 10-3	
	45	0.65	0.595	0.055	3.67 x 10-3	
	60	0.595	0.586	0.009	0.6 x 10-3	
NG2 Density-2.32	15	1.594	1.532	0.062	4.13 x 10-3	Sample breaks during handling, into small pieces
	30	1.532	1.496	0.036	2.4 x 10-3	
	45	1.496	1.463	0.033	2.2 x 10-3	
	60	1.463	1.444	0.019	1.12 x 10-3	
NG3 Density-1.55	15	1.618	1.097	0.521	34.7 x 10-3	Breaks in small pieces in first 15 days immersion
	30	1.097	1.03	0.067	4.47 x 10-3	
	45	1.03	1.0	0.03	2.0 x 10-3	
	60	1.0	0.975	0.025	1.67 x 10-3	
NG4 Density-1.48	15	1.639	1.39	0.249	16.6 x 10-3	(Start breaking) during handling along with white ppt. after 30 days immersion
	30	1.39	1.28	0.11	7.33 x 10-3	
	45	1.28	1.20	0.08	5.33 x 10-3	
	60	1.20	1.13	0.07	4.0 x 10-3	
NG5 Density-1.51	15	1.781	0.838	0.943	62.9 x 10-3	(Breaks in small pieces) light along with green ppt. in first 15 days immersion
	30	0.838	0.82	0.018	1.20 x 10-3	
	45	0.82	0.73	0.09	6.0 x 10-3	
	60	0.73	0.67	0.06	4.0 x 10-3	
NG6 Density-2.57	15	1.739	1.737	0.002	0.133 x 10-3	No breaking was observed
	30	1.737	1.728	0.009	0.6 x 10-3	
	45	1.728	1.721	0.007	0.467 x 10-3	
	60	1.721	1.714	0.007	0.467 x 10-3	

A testing of soil has been carried out to know the existing nutrient position before application of glass compositions prepared in the agricultural research field. For this purpose, soil sample was taken randomly at a depth of 15 cm and get it air dried. There after the soil sample was sieved through 2.0mm sieve packed in polythene bags and stored. By using DTPA extractable method with AAS for Fe, Zn, Cu, Mn, calorimetric method for B, available N by Kjeldahl method, and available P by Olsen's method and available k by Flame photometric method have been employed for carrying out the macro and micro elements analysis for the soil of our research field and related results are summarized in Table 4.

The field trial has also been conducted during the spring season in small research field which has been developed for testing the prepared glass compositions and their related observations are illustrated in Table 5. The glass compositions were applied on ladyfinger for seeding and plantations. By doing a comparative study among all the segments in terms of germination, plant height, plant branches and number of vegetative, it has been observed that among all six compositions only three compositions are giving water germinations (quick and more plants in numbers) rather than without ceramic and chemical nutrient segment. It is also evident from the table of agriculture parameters investigated that both the glass compositions show better and consistent results throughout the course of investigation and provide almost relatively similar and better response in comparison to chemical nutrients fertilizer used for study. While studying the experimental data observed from table 5, the effect of the vitreous fertilizers (NG4 for ladyfinger) show an evident influence along with maintaining their physiological functioning and nutrients deficiencies occur from time to time in agriculture fields.

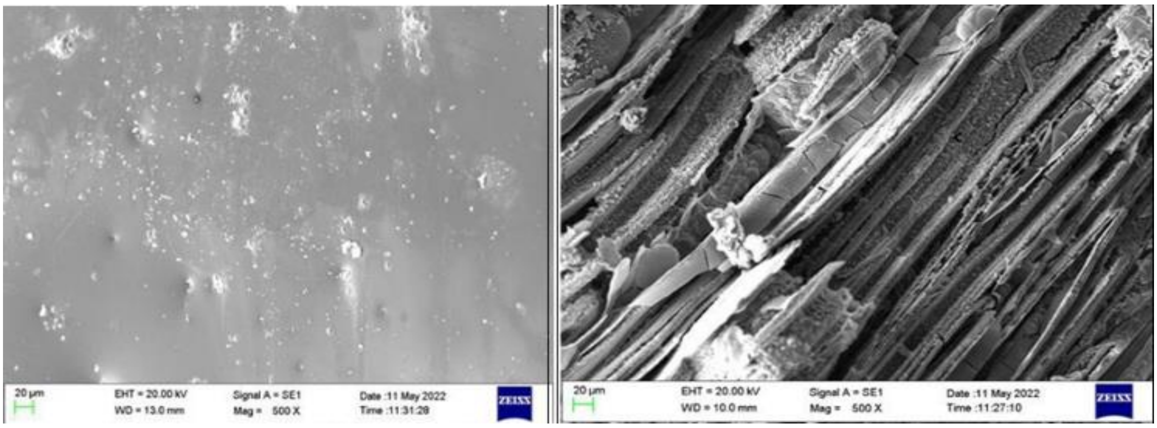

Fig 2: (a) SEM Image of NG4 Unleached (b) SEM Image of NG4Leached

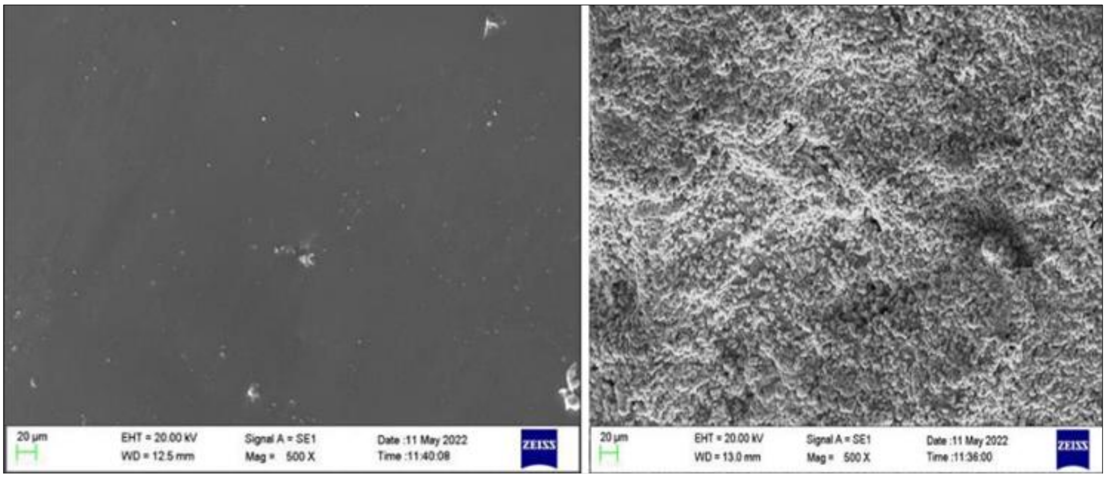

Fig 3: (a) SEM Image of NG5 Unleached (b) SEM Image of NG5Leached

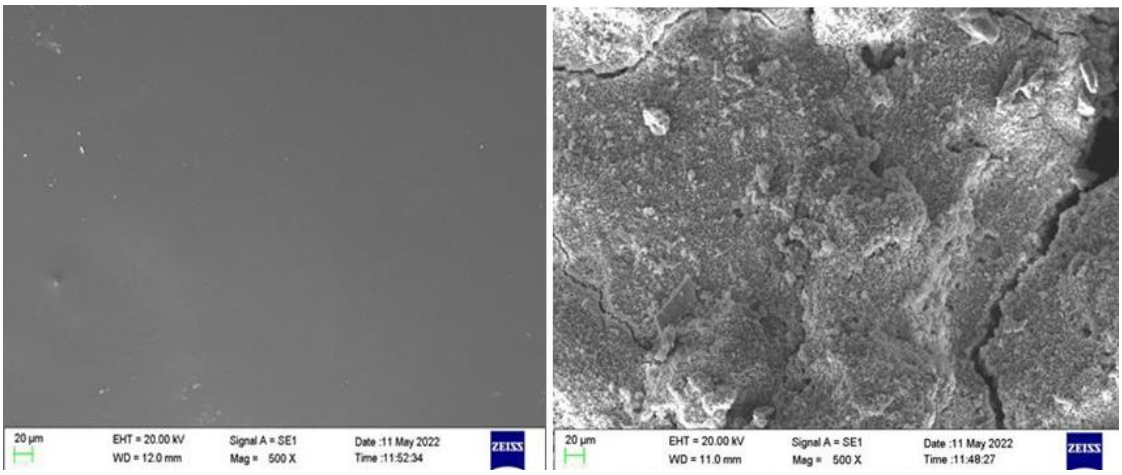

Fig 4: (a) SEM Image of NG3 Unleached (b) SEM Image of NG3Leached

Table 4: Macro and micro-nutrient elements concentration in (ppm) the soil observed along with pH and TDS

pH 7.94	TDS 390	Organic carbon%	N (kg/ha)	P (kg/ha)	K (kg/ha)	B (ppm)	Fe (ppm)	Cu (ppm)	Zn (ppm)	Mn (ppm)	S (ppm)
Observed Values		0.41	140.0	34.28	189.17	0.62	12.11	1.56	0.79	7.32	12.36
Std. Range		0.10-1.11	40-245	3-90	70-600	0.10-11.5	2-80	1-6.50	0.10-5.37	2-28	2-59

Table 5: Research field results of samples

Duration (in Days)	Plants Observation	Without Fertilizer	Chemical Fertilizer	Ceramic Fertilizer					
				NG 1	NG 2	NG 3	NG 4	NG 5	NG 6
5	Germination	low	good	low	good	good	good	good	low
10	Plant Height (cm)	6	8	7	8	8	8	8	7
	No. of plant branches	2	4	4	4	4	4	4	4
20	Plant Height (cm)	8	12	9	10	11	12	11	9
	No. of plant branches	3	5	5	5	5	5	5	5
30	Plant Height (cm)	11	17	14	14	15	17	15	13
	No. of plant branches	3	6	5	5	6	6	6	5
40	Plant Height (cm)	14	21	19	20	20	21	20	18
	No. of plant branches	4	6	5	5	6	6	6	5
50	Plant Height (cm)	16	24	21	22	22	24	22	20
	No. of plant branches	4	8	6	7	7	8	7	6
	No. of flowers/vegetates	1	3	2	2	2	3	2	2
60	Plant Height (cm)	19	27	23	25	25	28	25	22
	No. of plant branches	5	8	6	7	7	8	7	6
	No. of flowers/vegetates	2	4	3	3	3	4	3	3

Conclusions

The study of the glass nutrients was done to make an alternative of the chemical fertilize was a major reason for the depletion in the quality of the soil. The results produced during the experiment of the glass fertilizers clearly justify its superiority over chemical fertilizers in terms of durability and non-toxicity. The leachability results show that the important nutrients are leached out from the glass matrix in a slow and controlled manner. The process was also continuous and hence supports it durability purpose. The pH result didn't show any permanent increase or decrease in the pH of soil and the value of pH was moving more towards the alkaline atmosphere which is very supportive for the vegetation purpose. Apart from this the characterization used supported our basic objective of slow and continuous leaching. The change in absorption spectrum of different bonds clearly establishes the fact the leaching was going on. The release of elements from the matrix was based on the principle discussed in the literature review that release of cations will be in order of monovalent, divalent, trivalent and tetravalent respectively. The field test completely established the fact by the data produced that glass fertilizers are much better than the chemical fertilizers in term of production. The present work was successful in the preparation of glass powders to be used for enhancing food production and in maintaining soil fertility. Immersion test method showed that the leaching span was higher in the beginning and became slower in later course of investigation. Finally, a rate of leaching was observed for NG4 and its rate of leaching was throughout the course of study. Surface morphology of the glass samples also confirmed the results obtained during leaching experiments. The prepared glass compositions were successfully applied on the ladyfinger which provided a better germination along with other agriculture parameters investigated throughout the course of study. It is important to mention that both the tested glass compositions may be a better substitution of chemical fertilizers for enhancing the production as well as removal of non-fertility in Indian agricultural situations. On the basis of various studies, it is concluded among all six samples NG4 is having more effectiveness which has been supported by the data from their analysis of leaching behavior, density, SEM, FTIR analysis and agriculture field trials.

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