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Spectroscopic Characterization (AAS, XPS, UV) of Glass-Derived Fertilizers and Their Agronomic Potential in Sesame

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Abstract

The global population increased rapidly and was expected to reach about 16.46 billion by 2050. At the same time, the amount of arable land per person decreased, creating an urgent need to rethink fertilizer use and find innovative ways to ensure food security. To address this challenge, researchers developed phosphate-based glass fertilizers using a melt-quenching method. The process involved heating various batch compositions to around 750°C for 30 minutes to form a new class of fertilizers designed to enhance crop yield, improve nutrient efficiency, and reduce environmental harm through controlled nutrient release. The prepared glass fertilizers were thoroughly examined using advanced analytical methods such as atomic absorption spectroscopy (AAS), X-ray photoelectron spectroscopy (XPS), and ultraviolet spectroscopy (UV) to study their chemical and structural properties. Phosphate content was measured through a standardized gravimetric technique using quimocia reagent, ensuring precise and reliable results. To test their agricultural performance, controlled pot experiments were conducted on sesame plants. Growth parameters such as plant length, grain size, and grain weight were compared with those obtained using conventional fertilizers. The results, presented in graphical and tabular forms, showed that the glass fertilizers significantly improved crop growth and productivity. Overall, the study demonstrated that phosphate-based glass fertilizers had great potential to increase crop yield, enhance nutrient use efficiency, and reduce environmental pollution. These findings highlighted their promise in promoting sustainable agricultural practices that balance food production with environmental protection and social responsibility, while emphasizing the need for continued research and optimization of these innovative fertilizers.

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Keywords
AAS;
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Abbreviations

AAS	Atomic absorption spectrometry
GS	Glass fertilizer sample
UV	Spectroscopy- Ultraviolet-Visible Spectroscopy
XPS	X-ray photoelectron spectroscopy

Introduction

The exponential augmentation of the global populace precipitates a concomitant escalation of environmental pressures, thereby underscoring the imperative for judicious environmental stewardship in the face of burgeoning ecological exigencies. The rapid proliferation of the global population, which has undergone a significant augmentation of approximately 43% over the past three decades, from 5.76 billion in 1995 to 8.23 billion in 2025,[1] poses formidable challenges to sustainable agricultural practices, necessitating a paradigmatic shift in the way we approach food production and environmental sustainability. Over the last 30-year population growth shown in Fig. 1. The average cultivable land per capita (ha) in earth progressively diminishes every year which is shown in Fig. 2.

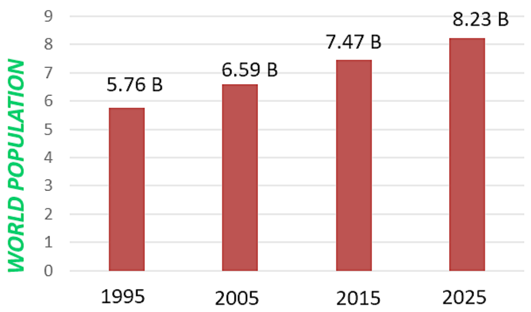

Fig. 1: World Population per 10 Years

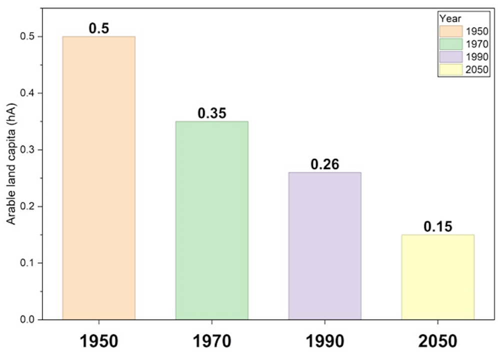

Fig. 2: A comparative study of arable land with time;[From: Phosphate Newsletter 23(2005)][2,3]

The paradoxical juxtaposition of escalating population pressures and diminishing arable land per capita precipitates a complex array of environmental and agricultural challenges, including soil degradation, water pollution, and loss of biodiversity.[2,4] The judicious application of fertilizers, comprising a diverse array of macro- and micronutrients, is crucial for enhancing plant growth and agricultural productivity, while minimizing the environmental impacts associated with fertilizer use.[1,4] The strategic deployment of fertilizers that exhibit rapid dissolution rates, minimal environmental persistence, and targeted nutrient delivery can help mitigate the risks associated with fertilizer use, while optimizing crop yields and promoting sustainable agricultural practices. Furthermore, the categorization of fertilizers into various types, including organic, inorganic, slow-release, and controlled-release formulations, affords farmers and agricultural practitioners a range of options for tailoring their nutrient management strategies to specific crop requirements and environmental contexts, thereby promoting a more nuanced and sustainable approach to agriculture.[5,6] The concomitant diminution of arable land per capita, coupled with the burgeoning global population, underscores the imperative for innovative solutions to ensure food security while minimizing environmental degradation. In this context, the development and deployment of novel fertilizers, such as glass fertilizers, can play a critical role in promoting sustainable agricultural practices and ensuring global food security.[7, 8]

Fertilizer is natural or chemical compounds that contain various crucial minerals. Such as Nitrogenium (N), Phosphoros (P), Kalium(K), Cuprum (Cu), Ferrum(Fe), Zincum (Zn), Calcium(Ca), Magnesium (Mg) etc. fertilizers assist the growth and development of plant or crops in agriculture.[10] It also helps to increase food production of plants. Again, the judicious application of fertilizers, which comprise a diverse array of crucial nutrients including extensive (macro) nutrients such as Nitrogen, Phosphorus, and Kalium, as well as micronutrients like copper, iron, zinc, and molybdenum, is crucial for optimizing plant growth and agricultural productivity. However, the indiscriminate use of these substances can have far-reaching environmental repercussions, including soil degradation, water pollution, and loss of biodiversity.[10, 11] To mitigate these risks, it is imperative to adopt a nuanced approach to fertilizer application, one that balances the need for enhanced crop yields with the imperative of environmental stewardship. This can be achieved through the strategic deployment of fertilizers that exhibit rapid dissolution rates, minimal environmental persistence, and targeted nutrient delivery, thereby minimizing the potential for ecological harm. Furthermore, the categorization of fertilizers into various types, including organic, inorganic, slow-release, and controlled-release formulations, affords farmers and agricultural practitioners a range of options for tailoring their nutrient management strategies to specific crop requirements and environmental contexts.[1-6] Bulk applications of inorganic fertilizer such as urea [$Co(NH_2)_2$] it releases mainly nitrogen (N). We know nitrogen shown detrimental effects on phreatic water character. Specifically, urea from nitrate ion in soil, which is deleterious for health. Furthermore, urea produces gaseous NH_3 and NO_x which is dangerous for eco system. So, we can say inorganic fertilization [$CO(NH_2)_2$] have pessimistic effects on the environment. Therefore, the main objective of agriculture is increasing production or yield with the minimal and adequate use of fertilizers.[7-11]

Table I: Composition of glass batches (in wt. %)

Composition of the prepared Glass Fertilizers (in wt. %)								
Glass ID	**$(NH_4)_2H_2PO_4$**	**$K_2H_2PO_4$**	**Borax**	**ZnO**	**MnO_2**	**Fe_2O_3**	**MoO_3**	**CuO**
GS-1	20	20	20	10	10	5	10	5
GS-2	20	20	20	8	10	5	12	5
GS-3	20	20	20	6	10	5	14	5
GS-4	20	20	20	4	10	5	16	5
GS-5	20	20	20	2	10	5	18	5

Materials and Methods

The glassy fertilizer samples are identified as GS. These glassy fertilizers consist of some essential element such as Nitrogen (N), Phosphorus (P), and Potassium (K), which are pivotal for crops growing. In addition, the sample includes base oxides and micro-element oxides necessary for plant development, including Iron (Fe), Zinc (Zn), Molybdenum (Mo), Boron (B), Copper (Cu), and Manganese (Mn), with each element exceeding 50 grams in quantity. Table I presents the oxide composition of GS in terms of wt. %. The glassy ingredients used in this study were prepared from various raw materials, including Ammonium dihydrogenorthophosphate [$(NH_4)H_2PO_4$], Magnesium Oxide (MgO), and Potassium dihydrogenphosphate (KH_2PO_4) which were used as the sources of macro-elements. To supply the necessary micro-elements, the following were added: Borax ($Na_2B_4O_7$), Ferric Oxide (Fe_2O_3), Zinc Oxide (ZnO), Molybdenum Trioxide (MoO_3), Manganese dioxide (MnO_2), and Cupric Oxide (CuO) all are AR & GR grade.Five glassy compositions were prepared using these precursor materials, as outlined in Table I. The raw materials for the glass batches were precisely weighed using a four-decimal electronic balance (Satorious, model BSA224SCW). All anhydrous and pure constituent were placed in agate mortar and mixed uniformly with a pestle for one hour. This mixing process was repeated three times to ensure consistency and homogeneity.

The glass batches were then melted in phosphate system, as outlined in Table I. The mixture of raw materials, prepared as described above, was dried and transferred into high alumina crucibles. These crucibles were then fired in a muffle furnace, equipped with a programmer, at a temperature range of 750-800°C for a soaking period of half an hour under ambient conditions. The actual melting process lasted for two hours. Throughout this operation, both the temperature and duration of melting were critical factors that required careful monitoring.The melting procedure took place in a programmable muffle furnace that was equipped with a window and temperature controller, allowing for adjustments to the temperature and melting time as needed. Table II provides the details of the melting conditions and time for each of the different glass batches.[4,12]

Table II: Melting point (°C), time of Soaking period and density for different glassy compositions

Glass ID	M.P (±5°C)	Soaking period (min)	Density(g/cc)
GS-1	800	30	1.4770
GS-2	750	30	1.4689
GS-3	750	30	1.4483
GS-4	760	30	1.4861
GS-5	750	30	1.4695

The glass structure and surface homogeneity of the melted batch mixtures were confirmed by the X-ray Powder Diffraction (XRD) and Scanning Electron Microscope (SEM) technique.[13]

The percent of the different elements within the prepared Glass Fertilizer sample were measured by the AAS by dissolving the definite quantity (0.1g) of the glass powder within the definite quantity (50ml) of HCl solution, which was further diluted with conductivity water. For the determination of Na, K, Mo and Mn the stock solution was diluted to (1:250) ratio with conductivity water and for the Fe, Cu and Zn the stock solution was diluted in the (1:100) ratio.[14] The sample concentration of the prepared solutions for the AAS elemental determination are shown in the Table III.

Table III: Preparation of sample solution for the AAS elemental determination

	Sample ID			
	GS-1	GS-2	GS-3	GS-4
Amount of glass sample (g) taken to prepare 50 ml soln.	0.1012 g	0.1676 g	0.1550 g	0.1406 g
Sample concentration	2024mg/L	3352mg/L	3100mg/L	2812mg/L
Sampleconcentration after (1:100) dilution	20.24 mg/L	33.52 mg/L	31 mg/L	28.12 mg/L
Sampleconcentration after (1:250) dilution	8.096 mg/L	13.408 mg/L	12.4 mg/L	11.248 mg/L

Now let us get for any analyte the instrumental value = y mg/l, so

% of Analyte= (y ml/l)/(x mg/l)×100

The spectrophotometric determination of phosphate, were done using the "molybdenum blue method," involves reacting a sample with ammonium molybdate in an acidic solution to form molybdophosphoric acid. This is followed by a reduction with a reducing agent like Sodium Sulfide and Potassium Antimony Tartrate, which produces a blue-coloured complex (molybdenum blue).[15, 16] The chemical reactions are as follows:

$$72H^+ + PO_4^{3-} + 12\ M0_7O_{24}^{6-} \rightarrow 7\ PMo_{12}O_{40}^{3-} + 36H_2O$$

$$PMo_{12}O_{40}^{3-} + 2C_6H_8O_6 \rightarrow PMo_{12}O_{40}^{7-} + 2C_6H_6O_6 + 4H^+$$

The intensity of this blue colour, which is proportional to the phosphate concentration, is then measured with a spectrophotometer at a specific wavelength (e.g., 830 nm).Molecular Weight of Complex, $P(MoO_3)_{12}O^4$ =1822.4;

$$Fraction\ of\ PO_4^{3-} = \frac{P + (4 \times 16)}{1822.4} = \frac{31 + 64}{1822.4} = 0.0521$$

$$\%\ of\ PO_4^{3-} = \left(\frac{UV - Vis\ data}{Sample\ Concetration\ under\ analysis}\right) \times 100 \times 0.0521$$

XPS was used for elemental analysis for quantitative analysis, which determines the elemental composition by measuring the number of photoelectrons emitted from a sample's surface, and chemical state analysis, which identifies the chemical bonding and oxidation state by analyzing the kinetic energy of these photoelectrons to determine their binding energy. Both methods involve irradiating the sample with X-rays and analyzing the resulting photoelectrons to identify elements and their chemical environment on the top few nanometres of the surface.[17-21]

Here, we have applied glass fertilizer in sesame (Sesamumindicum) cultivation through pot culture. Sesame of the Pedaliaceae family is one of the first oil seeds. Sesame oil is rich in protein and lipids and has much health advantage. At first, we take 6 pots then each tub filled with 10kg same type of soil. Then we plant 6 or 7 seed in each tub and numbering as 1, 2, 3,4, 5 and 6. Again we apply 1g of different glass fertilizer in each different tub as GS-1 in tub 1, GS-2 in tub 2, GS-3 in tub 3, GS-4 in tub 4, GS-5 in tub 5 and 1g urea usedin the last tub, tub 6.We are measured the growth i.e. height of the plants and number of grain and grain size present in each plant after 15 and 30 days of the cultivation. After harvesting we have taken the weight of 10 dry grains with respect to each tub.

Results and Discussion

XRD and SEM Studies

The glassy state of all the samples investigated was analyzed and confirmed using X-ray Powder Diffraction (XRD) technique as shown in Fig. 3, no crystalline peaks were observed, indicating that the synthesized glass samples are fully amorphous. The XRD patterns display only broad scattering peaks, known as the "halo" with an increased background in the lower range of the 2θ angle, which is a result of X-ray scattering.[5] The results presented in Fig. 3 are consistent with those previously published for other REO-doped phosphate glasses.[13]

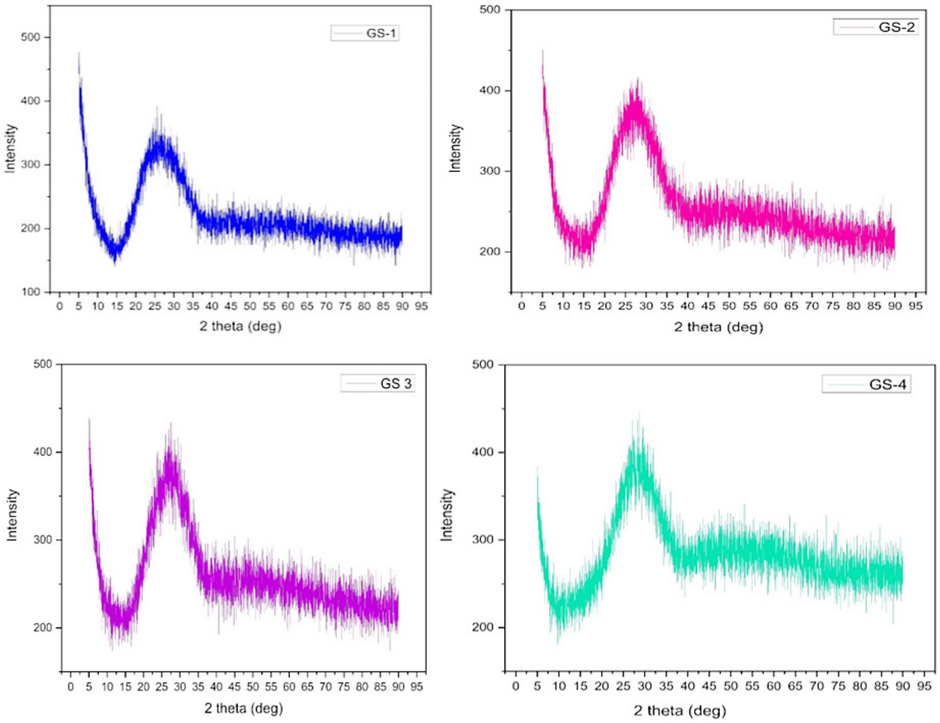

Fig.3: XRD Spectrum of glass fertilizer sample

SEM images of some glass fertilizer sample were performed which has been shown in Fig.4. The photograph indicate homogeneous nature of that the glasses formed.

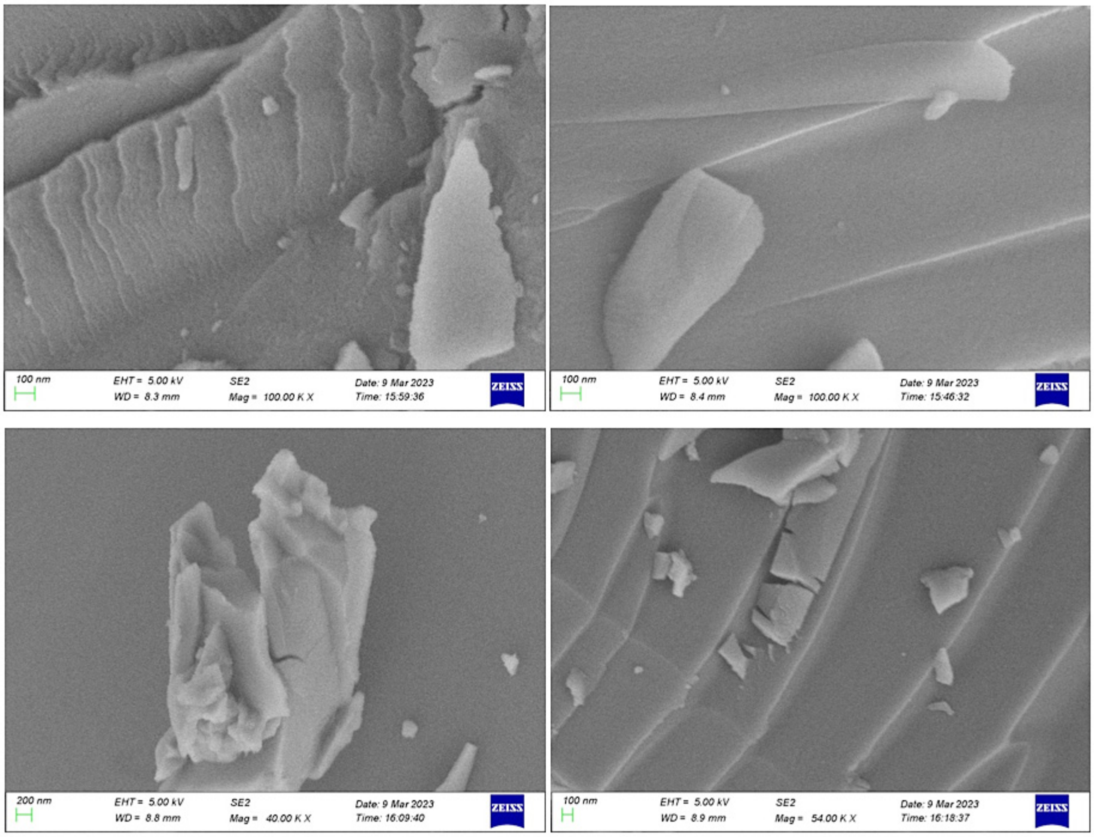

Fig.4: SEM structure of prepared sample GS-1, GS-2, GS-3, GS-4 (clock wise from left top).

The results of the elemental analysis in the prepared glass sample done by AAS analysis presented in Table IV. The result of the spectrophotometric determination of phosphate are shown in Table V.

Table IV: From AAS analysis data % composition of Na, K, Mo, Mn, Fe, Cu, Zn

Sample ID	Mass % of Na	Mass % of K	Mass % of Mo	Mass % of Mn	Mass % of Fe	Mass % of Cu	Mass % of Zn
GS-1	5.5014	5.5237	17.64	6.71	0.27	4.20	1.88
GS-2	4.4637	3.3353	21.44	5.98	0.28	4.68	1.42
GS-3	12.1064	7.1096	25.20	6.06	0.25	4.84	0.76
GS-4	13.0654	11.6153	35.38	11.66	0.38	9.42	1.52

Table V: % Composition of PO_4^{3-} in different Glass fertilizer sample

Sample ID	Sample weight(g) takento make 50ml of sample solution.	Conc. After (1:2500) dilution (mg /L)	PO_4^{-3} concentration as per Instrumental result (ppm)	% of PO_4^{-3} in the sample
GS-1	0.1012	0.8096	4.57	29.41
GS-2	0.1676	1.3408	9.32	36.22
GS-3	0.1550	0.31	5.07	84.82
GS-4	0.1406	0.2812	5.011	92.84

By XPS study we have determined atomic percentage of O, C, P and N, in the prepared glass fertilizer samples. In the Table-VI we are showing atomic percentage of O, C, P and N, in GS-1, GS-2, GS-3, GS-4, GS-5 samples. In Fig. 5(a-e) XPS plots of the same are shown respectively.

Table VI: % composition of O,C,P and N from XPS study

Sample ID	% of O	% of C	% of P	% of N
GS-1	62.2	22.0	8.5	4.2
GS-2	56.8	24.6	8.7	8.0
GS-3	52.2	32.2	6.8	6.7
GS-4	60.0	20.0	11.2	8.8
GS-5	60.2	18.4	11.0	7.4

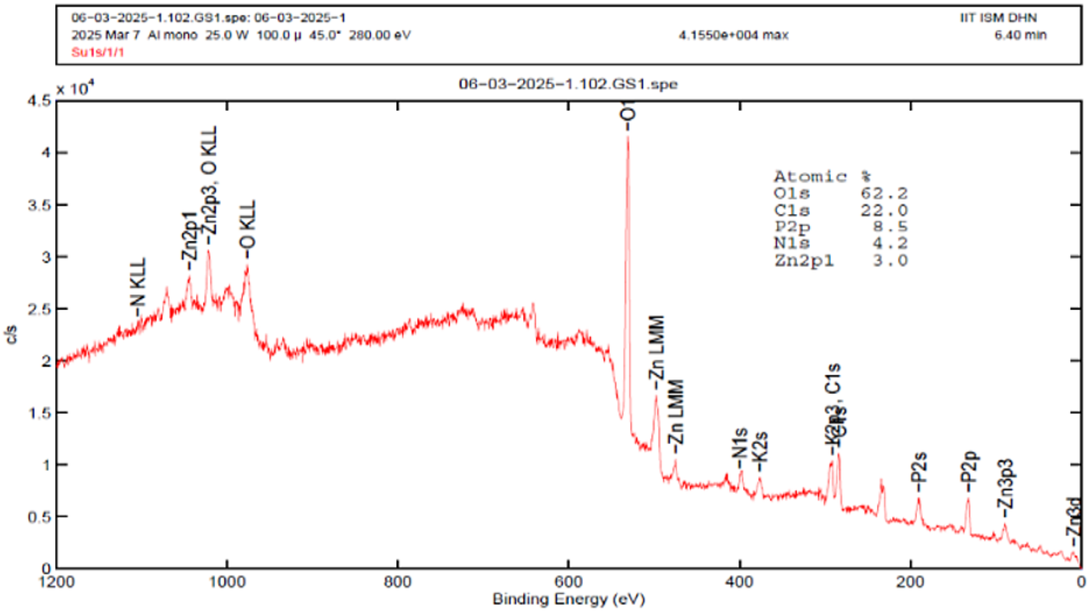

Fig. 5(a): XPS plot of GS-1

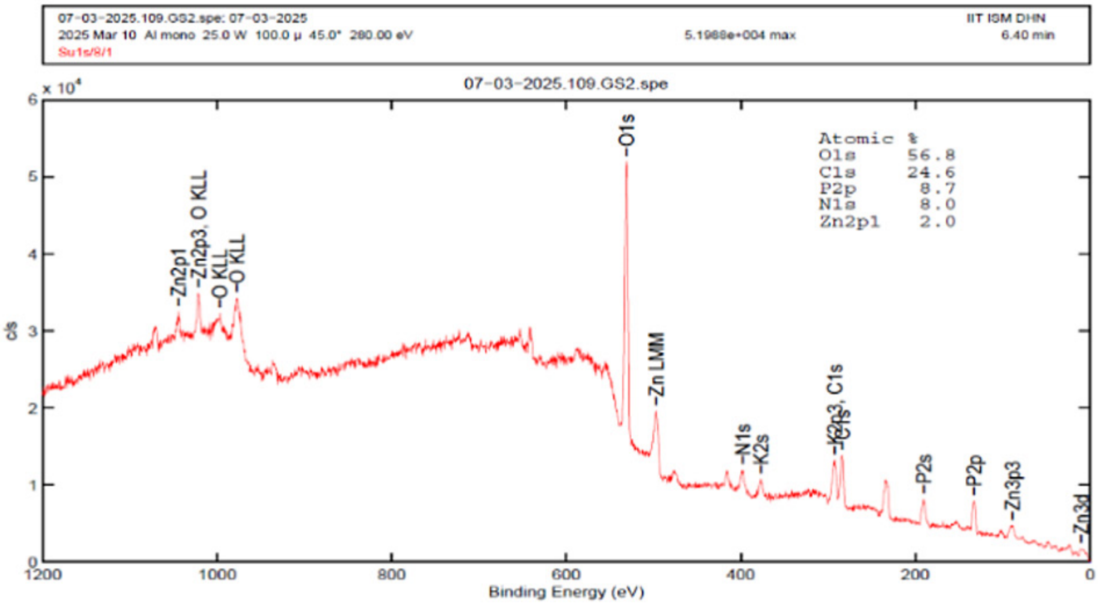

Fig. 5(b): XPS plot of GS-2

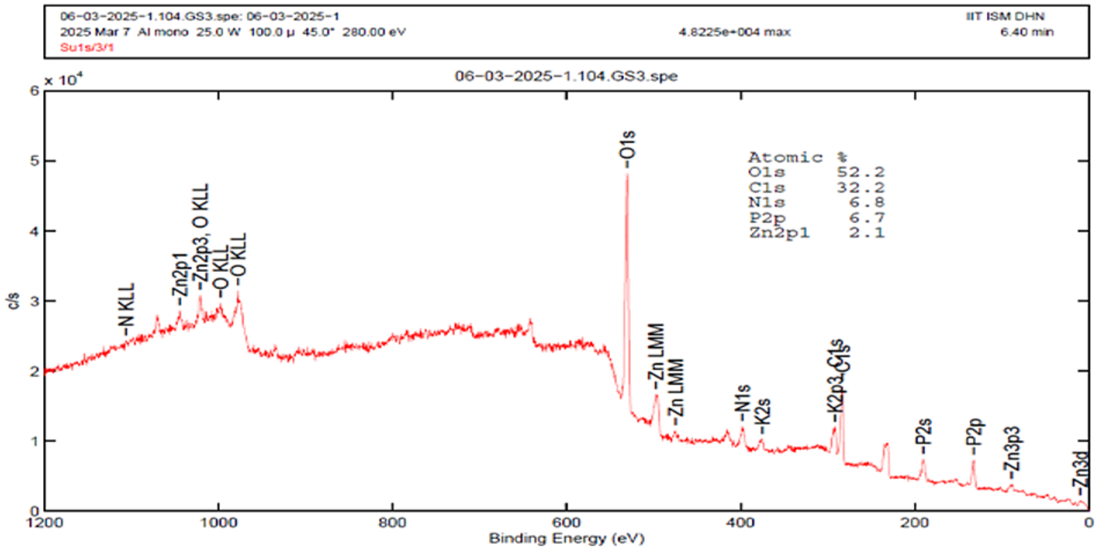

Fig. 5(c): XPS plot of GS-3

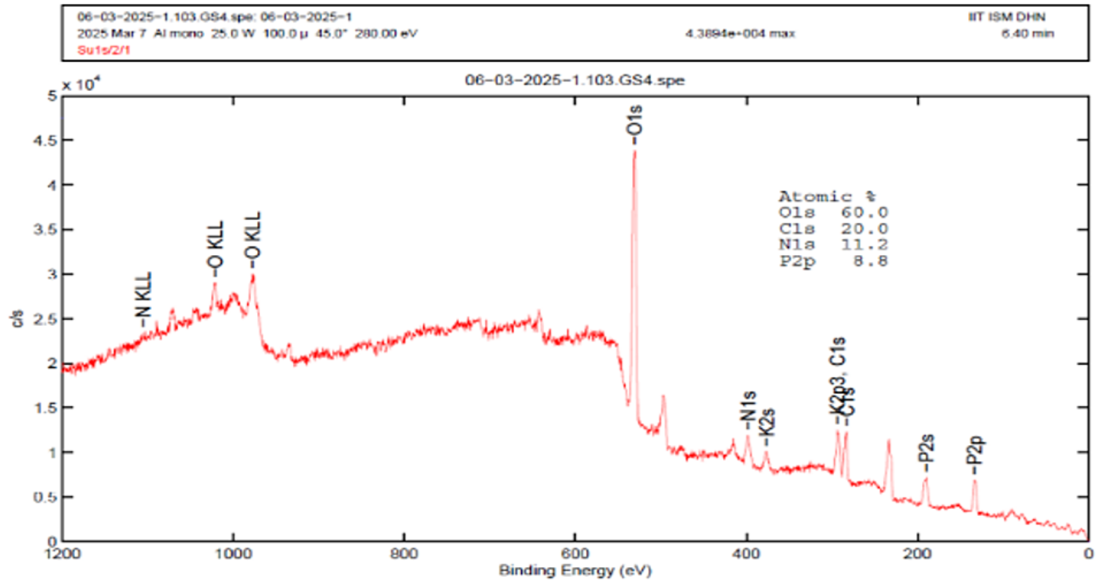

Fig. 5(d): XPS plot of GS-4

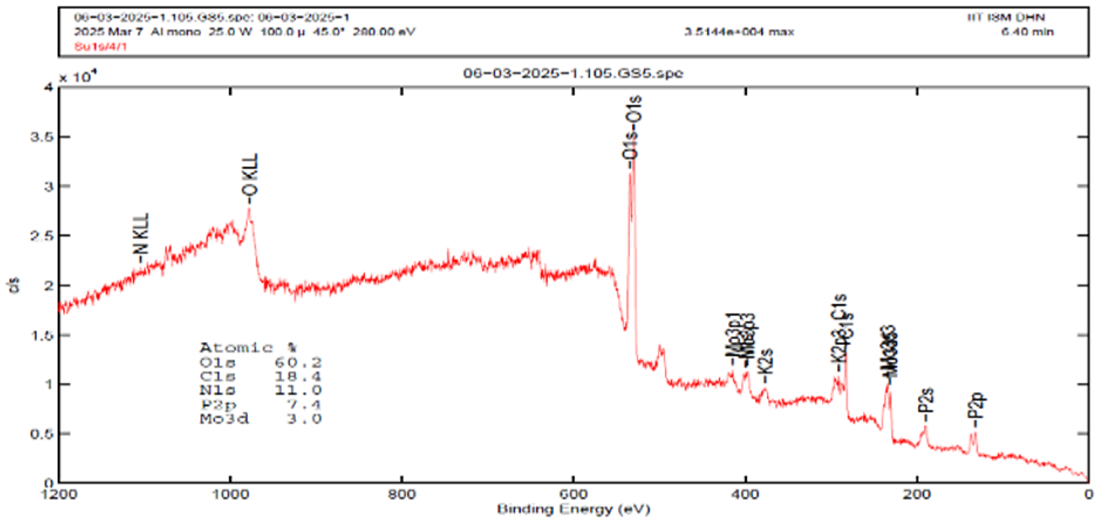

Fig. 5(e): XPS plot of GS-5

The results of the different Glass Fertilizers applications in thi Sesame cultivation under pot culture are observed time to time. The heights of the sesame plants have been measured and number of grain appeared in each plants were counted which are shown in Table VII. The Table VIII presents the average height and average number of grain. Again the grain's size and grain's weight were measured which are presented in the Table IX. Table X represents average grain's size and average grain's weight. After harvesting total weight of the Sesame seeds obtained from 10 dry grains taken from each tub are presented in Table XI.

Table VII: The height of the plants and number of grain present in each plant

Serial No	GS-1		GS-2		GS-3		GS-4		GS-5		Without GS	
	Height (inch)	No of grain	Height (inch)	No of grain	Height (inch)	No of grain	Height (inch)	No of grain	Height (inch)	No of grain	Height (inch)	No of grain
1.	27	16	20	07	21	09	26	11	22	08	19.5	03
2.	24	12	16	05	20	09	25	12	21	07	17.5	05
3.	20	09	21	08	18	07	16	07	19.5	07	16.9	08
4.	23	11	18	06	16	08	19	09	17.5	05	17.4	05

Table VIII: Average height and number of average grain from each plant

GS-1		GS-2		GS-3		GS-4		GS-5		Without GS	
Average Height (inch)	No of average Grain	Average Height (inch)	No of average Grain	Average Height (inch)	No of average Grain	Average Height (inch)	No of average Grain	Average Height (inch)	No of average Grain	Average Height (inch)	No of average Grain
23.5	12	18.75	6.5	18.75	8.25	21.5	9.75	20	6.75	17.82	5.25

Table IX: Grain size and its weight of 10 grains

Serial No.	GS-1		GS-2		GS-3		GS-4		GS-5		Without GS	
	Grain Size (Cm)	Grain Weigh (mg)	Grain Size (Cm)	Grain Weigh (mg)	Grain Size (Cm)	Grain Weigh (mg)	Grain Size (Cm)	Grain Weigh (mg)	Grain Size (Cm)	Grain Weigh (mg)	Grain Size (Cm)	Grain Weigh (mg)
1.	2.9	632.5	2.9	601.0	2.7	501.1	3.2	528.6	2.9	617.5	2.6	522.2
2.	3.3	623.6	2.6	662.0	2.8	523.0	2.9	501.5	2.9	557.8	2.3	425.1
3.	3.3	635.5	2.6	630.7	2.8	567.0	3.2	568.2	3.1	604.0	2.3	430.4
4.	2.7	523.1	2.8	572.8	3.0	555.8	3.3	601.3	3.3	670.1	2.4	413.5
5.	2.8	555.9	3.0	589.6	2.9	531.7	3.2	624.6	3.0	508.6	2.2	268.7
6.	3.1	720.7	2.7	716.3	2.7	557.3	3.2	578.3	3.0	612.2	2.6	493.1
7.	3.0	657.5	2.8	654.9	2.9	582.1	3.4	633.6	2.8	600.5	2.5	487.4
8.	3.0	643.5	2.7	559.8	2.8	533.2	2.9	553.1	3.0	569.4	2.6	529.6
9.	2.9	746.9	2.4	404.8	2.5	451.8	2.6	507.1	3.1	571.1	2.1	432.9
10.	3.3	683.8	2.7	567.7	2.4	458.1	3.0	507.1	2.9	645.4	2.5	487.6

Table X: Average grain's size and average grain's weight

GS-1		GS-2		GS-3		GS-4		GS-5		Without GS	
Average Grain size (cm)	Average Grain Weigh (mg)	Average Grain size (cm)	Average Grain Weigh (mg)	Average Grain size (cm)	Average Grain Weigh (mg)	Average Grain size (cm)	Average Grain Weigh (mg)	Average Grain size (cm)	Average Grain Weigh (mg)	Average Grain size (cm)	Average Grain Weigh (mg)
3.03	642.30	2.72	595.99	2.75	526.11	3.09	560.36	3.00	595.66	2.41	449.05

Table XI: Total Weight of the Sesame seedsgot from 10 dry grains after harvesting from the each Tub

Total 10 Dry Grain weight (g) after harvesting from the each Tub					
GS-1	GS-2	GS-3	GS-4	GS-5	Without GS
1.6769	1.3850	1.2292	1.5661	1.4949	0.8637

It has been observed that the growth and development of the sesame plants on the tubs were better on which glass fertilizers (GS) were applied. Among all the pots, tub no. 1 & 4 was maximum growth and production in which GF-1 & GF-4 glass fertilizers were applied and the tub no.6 was minimum growth and yield where no glass fertilizer was used.

Conclusion

In conclusion, this research provides compelling evidence of the transformative potential of phosphate-based glass fertilizers in advancing sesame cultivation while maintaining a balance between agricultural productivity and environmental sustainability. The combined insights from UV, AAS, and XPS analyses confirm the effectiveness of these fertilizers, demonstrating their capacity to significantly enhance sesame yield, nutritional value, and overall agronomic performance. Given the crucial role of sesame in global food security, sustainable farming systems, and climate-change mitigation, these findings highlight glass fertilizers as a promising and innovative solution.Moreover, the outcomes of this study hold significant implications for the future of sustainable agriculture, emphasizing the role of advanced fertilizer technologies in reshaping modern farming practices. The adoption of glass fertilizers can help optimize crop production, reduce environmental harm, and support essential ecosystem services, thereby contributing to a more resilient, sustainable, and food-secure world. As global population growth intensifies pressure on food systems, the ability of glass fertilizers to improve crop yield, nutritional quality, and environmental sustainability becomes increasingly vital, underscoring the need for continued research and development in this emerging field.

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Conflict of Interest

The authors do not have any conflict of interest.

Data Availability Statement

The manuscript incorporates all datasets produced or examined throughout this research study.

Ethics Statement

This research did not involve human participants, animal subjects, or any material that requires ethical approval.

Informed Consent Statement

This study did not involve human or any others animals participants. Therefore, informed consent was not required.

Clinical Trial Registration

Not Applicable.

Permission to Reproduce Material from Other Sources

Not Applicable.

Author Contributions

- **Gouranga Saha:** Conceptualization, Methodology, Data Collection, Analysis, and draft writing.
- **Dr. Goutam Hazra:** Conceptualization, Methodology, Writing and Editing of the manuscript, Communication with the journal's editor for publication.
- **Dr. Tanmoy Das:** Conceptualization, Methodology, Visualization and Supervision of the total work.

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