

## Nano-silica Synthesis, Characterization and Their Effects on Rice Production in the Presence of Calcium Humate under Salinity Soil Stress

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**Abstract:** Agricultural by-products have been repurposed to manufacture specific compounds through cost-effective technologies in order to maximize their use. These artificial substances are being utilized to augment the capacity of rice plants to tolerate salinity, thereby increasing their productivity. So, rice husk was used to synthesize Nano-silica (NSi), which was characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM), while calcium humate (CaH) was extracted from compost. Subsequently, a field experiment on saline soil was carried out at Sahl El-Hossinia Agric. Res. Station Farm in El-Sharkia Governorate, Egypt. The experiment was conducted throughout the summer season of 2022 to investigate the influence of NSi, CaH and their combination NSi+CaH at various concentrations on rice crop productivity and soil properties. Three NSi treatments (2, 3 and 4 mM) and other CaH levels (2,3 and 4 % as well as their combinations) were tested in the experiment. The study reveals that the addition of NSi significantly increased the biological, straw and grain yields of rice crops by 45, 52 and 34%, respectively. Similar results of CaH increased biological, straw and grain yields of rice with 7649, 4419 and 3229 kg fed<sup>-1</sup>. The NSi<sub>2</sub>+CaH<sub>2</sub> composite application produced superior yields. The total content of N, P, K and Si in the straw and grains of the rice increased as compared to the control. Also, NSi or CaH reduced SAR and ESP, but the NSi+CaH treatment had no significant reduction. All treatments significantly improved the availability of N, P, K and Si in the soil compared to the control treatment. In conclusion, this study shows that individual applications of NSi or CaH can effectively mitigate salinity stress by reducing sodium toxicity more than their combination.

**Key word:** Nano silica • Calcium humate • Salinity stress • Rice crop • Soil properties

### INTRODUCTION

Large amounts of agricultural waste can have a negative impact on the environment and ecosystems, such as greenhouse gas emissions, the formation of disagreeable odors and dangerous compounds that can enter water sources [1]. Rice husk (RH) is a byproduct of rice milling that accounts for approximately 20% of the weight of unhulled rice. Rice waste materials, as stated by [2], are one of the agricultural wastes that are increasing year after year. Therefore, rice waste is an appealing renewable resource for the production of nanomaterials as an alternative to harmful chemicals. In some countries, where rice is the main crop consumed by the bulk of the population, this amount will be abundant. Consequently, managing the RH is necessary to obtain high-value products and lessen the environmental issues it causes.

In an effort to address the issue of this agricultural waste's availability, silica extraction from RH has been the subject of multiple studies published in recent years [3]. Silicon (Si) is a necessary component for plant growth. It is generally known that Si can promote plant development under salt stress. Due to silicon, certain plant species, including wheat, barley and rice, is thought to be less salt resistant. Several hypotheses have been proposed for Si-mediated salt stress alleviation, including [1] the buildup of Na in rice and barley shoots following Si addition under salinity stress. [2] Reduced Na absorption at the root [4, 5].

Also, nanoparticles (NPs) are particles with a size in at least one dimension ranging from 1 to 100 nm. When compared to bulk materials, they can affect physio-chemical properties more favorably since their increased surface area enhances solubility and surface reactivity [6].

Nano-silica (NSi) is one of the practical nanomaterials that has been shown to benefit current agriculture. NSi has been demonstrated to be advantageous in modern agriculture. Currently, however, there is a lack of understanding of how NPs affect plant growth and development [7].

Moreover, humic substances (HS) are the remains of broken-down plant and animal components such as cellulose and lignin [8]. High concentrations of HS are discovered in the soil following the integration of collected residues [9]. The most typical commercial sources of HS include soils, coal, lignite and organic materials [10]. While there are many different functional groups in the HA structure, the most common ones are the phenolic (OH) and carboxylic (COOH) groups. These groups are mostly in charge of HA actions, including improving the physical and chemical properties of soil [11, 10]. Furthermore, these functional groups allow it to form complexes with ions such as  $Mg_2^+$ ,  $Ca_2^+$ ,  $Fe_2^+$  and  $Fe_2^+$  [12]. Adsorption of cations or metals by HA can occur by (a) direct adsorption ( $Ca_2^+$  releasing  $PO_4^-$ ), (b) complexation of  $Cu_2^+$  or external field interactions of aqueous  $Mg_2^+$  and (c) the creation of a cation bridge via direct or indirect chelation [13]. These functional groups, in addition to dissociating, form polar and nonpolar ends, which are the hydrophilic and hydrophobic portions, respectively [14]; both ends play roles in the processes that impart desired HA activities. Also, humic acids promote crop development by increasing plant growth hormones such as auxin and cytokinin, both of which aid in stress resistance, nutrient metabolism and photosynthesis [15].

Calcium (Ca) is a crucial plant nutrient that protects plants from adverse weather conditions, controls cell wall formation and regulates growth and development [16]. It is essential for salt stress tolerance [17], promotes antioxidant enzyme activities, reduces lipid peroxidation during abiotic stress [18], maintains cell membrane surfaces, maintains cell pH and prevents solute escape from the cytoplasm [19]. According to [20], cytosolic Ca concentration is an essential intracellular messenger that synchronizes response to a range of developmental signals and environmental stresses. It also serves as a buffer for inorganic and organic anions in the vacuole. In a number of crop species, calcium also lessened crop damage brought on by stress [21]. The interaction of  $Na^+$  and  $Ca_2^+$  on plant development is well established and  $Ca_2^+$  preserves  $K^+$  transport and  $K^+/Na^+$  selectivity in  $Na^+$  challenged plants [22]. Thus, the application of calcium humate resulted in better plant growth and soil characteristics under salinity stress.

On the other hand, salinity is a major problem that affects food security and agricultural productivity globally (in irrigated deserts and semi-arid land). Salt has damaged 1-10 billion hectares of soil in more than 100 nations, with an annual increase rate of 10% [23]. In irrigated dry regions, where 20-50% of the land surface is affected by salt, soil salinization is more common [24]. Climate change is expected to accelerate the rate of expansion of territories affected by soil salinization [25]. Plants struggle to generate the soil firmness and hardness required for effective root systems when subjected to saline stress. Furthermore, salinity can be caused by a lack of water, which creates osmotic stress and reduces the efficiency with which minerals like nitrogen, phosphate, potassium and calcium are delivered, resulting in nutritional deficiency and ionic toxicity [26].

Salinity and silicon are crucial in higher plants, but little is known about the potential benefits of NSi in decreasing and relieving salt stress damage in plants [27]. The rice-growing system is highly susceptible to salt stress, which could impact rice productivity due to the growing population and increasing food demand. Rice is essential for global food security, with half of the world's population daily consuming it [28]. Saline soil affects rice plant growth, yield and nutritional value, leading to an ion imbalance that lowers rice quality [29]. Also, [30] show that rice cultivars' shoot and root tissues are susceptible to sodium salts, causing shoot length to decrease. Although  $Na^+$  has the same positive charge as  $Na^+$ , it has different functions in cells. A low concentration of  $Na^+$  in roots can partially fill in for insufficient  $K^+$  in the crop, while elevated  $Na^+$  levels in the cytosol are harmful.

Most previous studies examined the effect of NSi on rice plant development under salt stress, but only a few investigated the effect of NSi mixed with specific organic compounds (CaH) on plant growth under salt stress. So, the goal of this study was to synthesize and characterize NSi extracts from rice husks. Then investigate the effectiveness of NSi and CaH with different concentrations in reducing the detrimental impacts of soil salinity on rice plant growth, yield components and mineral total content, as well as some chemical soil properties.

## **MATERIALS AND METHODS**

Nanomaterials and the expanding nanotechnology sector have grown rapidly in recent years and nanomaterials are now widely used in many fields, including the environment and food production. Nano-silica synthesis from rice husk and characterization

by XRD and TEM were performed. Also, a field experiment was used to assess the effects of foliar applications of NSi and CaH with different concentrations and their combinations on rice crop production under saline soil conditions.

**Nano-silica Extraction:** Firstly, silica was extracted using a technique adapted from earlier research [31, 32]. The extraction of NSi from rice husk (RH) consists mostly of four processes, which are detailed below:

**Washing and Drying:** The RH is first washed with water to eliminate pollutants such as dust, sand and so on. After that, it is dried in an oven at 110 degrees Celsius for 12 hours (or until entirely dry). The dried RH is treated with 1 M HCl and stirred for 90 minutes. This RH is then decanted and washed with warm distilled water to remove any acid (test the water from washing the RH with silver nitrate) before being heated in an oven at 110°C for 12 hours (or until fully dry).

**Burning:** A known quantity of RH produced from the preceding procedure is burned at 700°C for 2.5 hours, yielding white-colored rice husk ash (RHA). The purpose of this phase is to decrease the material in order to improve the silica content of the samples and to burn off any unwanted components.

**Extraction of Silica:** In a beaker, 10 g of RHA is agitated with 50 mL of NaOH (2.5 M) solution for 3 hours, followed by filtration (or centrifugation until separated). The resulting residue is rinsed with 20 mL of boiling distilled water. After that, 10 M H<sub>2</sub>SO<sub>4</sub> is added to adjust the pH to 2, followed by the addition of NH<sub>4</sub>OH to adjust the pH to 8. After washing the precipitate (silica gel) with warm water and drying it in an oven, white silica is produced.

**Extraction of Nano-Silica:** Finally, HCl 6 M is added to the extracted silica, agitated for 4 hours and washed to eliminate the acid. 2.5 M NaOH (80 mL) is added and stirred for 1 hour. This solution is treated with 10 M H<sub>2</sub>SO<sub>4</sub> to obtain a pH of 8 (or until a white precipitate (Nano-silica gel) forms). The precipitate is rinsed with warm distilled water and baked in an oven for two days to produce white Nano-silica. We would like to highlight the rice husk components employed in the creation of Nano-silica using a schematic of Nano-silica synthesis (Fig. 1).

Extraction and purification were carried out in accordance with equations 1 and 2, as given by [33, 34].

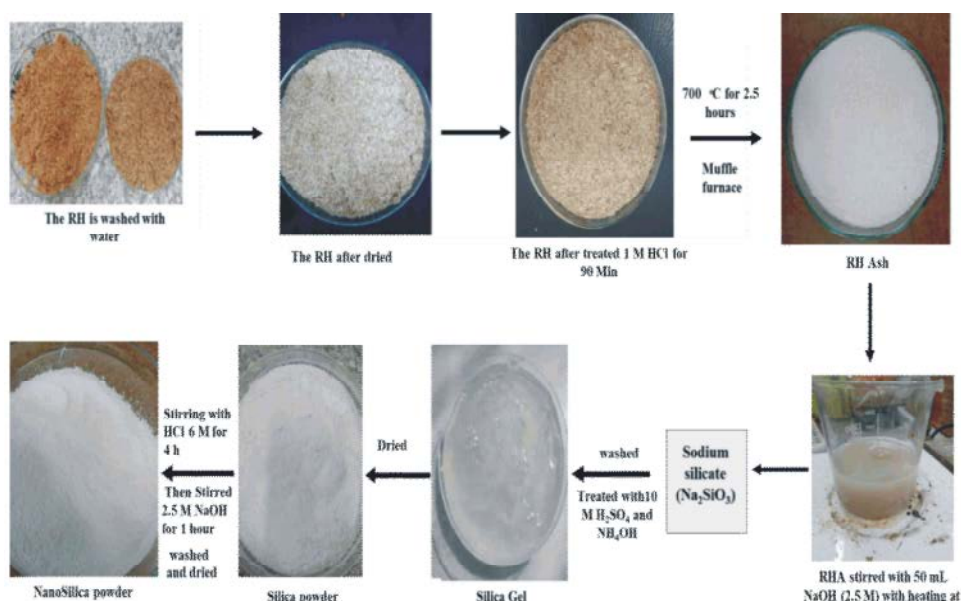
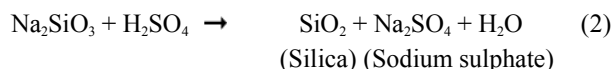
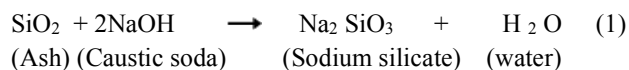


Fig. 1: Flow diagram of Nano-silica synthesis

**Nano-silica Characterization**

**1- X-Ray Diffraction (XRD) Pattern:** Only a few methods, such as X-ray diffraction (XRD) (X'Pert Pro, Pan Alytical, Netherlands) utilizing Cu K (= 1.5406) as a radiation source throughout the 2 range of 10°-80° at 293 K, may be utilized to analyze the crystalline nature of the generated Nano-silica particles.

**Transmission Electron Microscopy (TEM):** Transmission electron microscopy (TEM, Tecnai G20, FEI, Netherlands) was used to assess the shape and particle size of the produced Nano silica powder. A drop of the solution was deposited on the carbon-coated copper grids for TEM investigation and dried by allowing water to evaporate at room temperature. At 70 kV, electron micrographs were taken using a GEOL GEM-1010 transmission electron microscope.

**Field Trial:** A field experiment in clay soil was conducted at the Sahl El-Hossinia Agric. Res. Station Farm in EL-Sharkia Governorate, Egypt, the coordinates for the farm is located at Latitude 31° 8' 12.461" N and longitude 31°52' 15.496" E. To investigate the effect of foliar application of Nano-silica (NSi) and calcium humate (CaH) with different concentrations, as well as their interactions (NSi+ CaH), on rice (*Oryza sativa*) crop productivity and soil chemical properties under saline soil conditions. Table 1 presents some physical and chemical properties of the studied soil using the approach described by [35]. This experiment was irrigated by water from El-Salam canal. The chemical analysis of the irrigated water is shown in Table 2.

Table 1: Physio-chemical parameters of the experimental soil (0-30 cm) before cultivation

Particle size distribution (%)	Parameter	Value			
Coarse sand	4.71	pH*	8.12		
Fine Sand	10.02	EC(dS m <sup>-1</sup> )**	11.8		
Silt	35.12	Soil organic matter (%)	0.82		
Clay	52.15	SAR	13.4		
Texture grade	Silt clay	ESP	15.9		
Soluble cations and anions (meq L <sup>-1</sup> )		Available nutrients (mg kg <sup>-1</sup> )			
Ca <sup>++</sup>	30.2	CO <sub>3</sub> <sup>-</sup>	Nd	N	101
Mg <sup>++</sup>	40.3	HCO <sub>3</sub> <sup>-</sup>	10.5	P	3.5
Na <sup>+</sup>	79.1	CL <sup>-</sup>	88	K	298
K <sup>+</sup>	1.21	SO <sub>4</sub> <sup>-</sup>	51.8	Si	199

\*1:2.5 (soil: water suspension) \*\* EC soil past extract

Table 2: Chemical analysis of irrigation water

Character	value	Soluble cations and anions (meq L <sup>-1</sup> )			
pH	7.9	Ca <sup>++</sup>	3.5	CO <sub>3</sub> <sup>-</sup>	nd
EC (dS m <sup>-1</sup> )	1.8	Mg <sup>++</sup>	5.5	HCO <sub>3</sub>	4.3
EC (ppm)	1152	Na <sup>+</sup>	8.1	CL <sup>-</sup>	7.5
SAR	3.8	K <sup>+</sup>	0.4	SO <sub>4</sub> <sup>-</sup>	5.7

The experiment was conducted on rice (*Oryza sativa* L. Giza 178) through the summer season of 2022. The experimental treatments were arranged in a split-plot design with three replicates. Treatments included control, NSi, CaH and NSi+CaH, which were allocated as main plots and three concentrations of the treatments occupied the sub-main plots, as shown in Table 3. Foliar application of treatments was done two times, 30 and 60 days after sowing. Also, calcium humate was analyzed and its chemical properties are presented in Table 4.

**Fertilization:** During soil preparation, the soil was fertilized with P at a rate of 100 kg fed<sup>-1</sup> as superphosphate (15% P<sub>2</sub>O<sub>3</sub>) prior to sowing. Nitrogen was supplied to rice in the form of ammonium sulfate (20% N) at rates of 200 kg fed<sup>-1</sup> after 15, 30 and 60 days of planting. Potassium was administered in the form of potassium sulfate (48% K<sub>2</sub>O) at a rate of 50 kg fed<sup>-1</sup> in two equal doses at planting and 30 days after sowing.

**Measurements**

**Yield Components of Rice:** The biological yield was calculated by harvesting the complete plot. After separating the above-ground part, the grain and straw yields were collected. Straw and grain of rice crop representing each analyzed treatment were oven dried at 70°C for 48 hours, then crushed in a stainless mill before being digested using a combination of sulfuric acid and hydrogen peroxide according to the process reported by [35]. Finally, N, P, K, Si and Ca percentages were determined as described by [36, 37].

**Soil Analysis:** At harvest time, soil samples were taken from each experimental unit (plot). As stated by (38), soil samples were air dried, smashed, passed through a 2 mm sieve and stored in polyethylene bags for subsequent chemical investigation. According to [39], the available Si was extracted with 0.5 M acetic acid and calorimetrically identified as yellow silicomolybdic acid. The sodium adsorption ratio (SAR) and the exchangeable sodium percentage (ESP) were computed using the computation stated by [40]:

$$SAR = Na^+ / \sqrt{([Ca^{2+}] + [Mg^{2+}]) / 2}$$

$$ESP = 1.95 + 1.03 \times SAR$$

where Na<sup>+</sup>, Ca<sup>2+</sup> and Mg<sup>2+</sup> were expressed in meq L<sup>-1</sup>

Table 3: Experimental treatments

Control recommended does NPK					
Nano-silica (mM)		Calcium humate (%)		Nano-silica (mM) + calcium humate (%)	
NSi <sub>1</sub>	2	CaH <sub>1</sub>	2	NSi <sub>3</sub> +Ca H <sub>1</sub>	4 NSi+2CaH <sub>1</sub>
NSi <sub>2</sub>	3	CaH <sub>2</sub>	3	NSi <sub>2</sub> +Ca H <sub>2</sub>	3 NSi+3 CaH <sub>2</sub>
NSi <sub>3</sub>	4	CaH <sub>3</sub>	4	NSi <sub>1</sub> +Ca H <sub>3</sub>	2 NSi+4CaH <sub>3</sub>

Table 4: Chemical analysis of calcium humate applied.

Parameters	Values	Macro and Micronutrients (mg <sup>l</sup> )			
pH	8.00	N (%)	1.40	Mg	240
Organic carbon %	0.58	P	14.6	Fe	2.90
Organic matter (%)	0.998	K	0.90	Mn	0.10
C:N ratio	0.42	Na	0.99	Zn	-
		Ca	6000	Cu	0.10

**Statistical Analysis:** All data were statistically analyzed using the approach outlined by [41]. The differences between treatments were determined using the Least Significant Differences (LSD) test at the 0.05 probability level. Finally, all statistical analyses were carried out using the "MSTAT-C" computer software program, as described by [42]. The correlation coefficient (R) was obtained obtained using the Microsoft Excel

## RESULTS AND DISCUSSION

**Characterization of Nano-silica:** The morphology and functional groups of the synthesized NSi were characterized and it was confirmed to be nanometer-sized, as mentioned in earlier studies [43]. The purity, functional groups and particle size of the prepared NSi particles were structurally and morphologically characterized.

**X-Ray Diffraction (XRD) Pattern:** XRD was used to characterize Nsi derived from rice husks. The XRD pattern of Nsi extracted from silica at 700°C is shown in Fig. 2.

The XRD pattern of NSi, which has a significant broad peak centered at a 20°-22° angle, verifies silica's amorphous nature. These findings are comparable with previous research that recovered silica nanoparticles from rice husk and showed a large peak centered at a 2° angle of 22° [31, 44, 45]. As a result, it is possible to conclude that NSi is entirely amorphous. Furthermore, at additional scanning angles ranging from 10° to 80°, no sharp peak was found, demonstrating the absence of any organized crystalline structure.

### Transmission Electron Microscope (TEM) Analysis:

A transmission electron microscope (TEM) was used to collect information on particle shape and size. Fig. 3 shows a typical TEM micrograph of NSi. The NSi particle size was obtained on the nanometer scale, with particles being spherical and having a diameter in the range of 4.37-20 nm, as shown in Fig. 3. This finding is consistent with prior research by [34, 46].

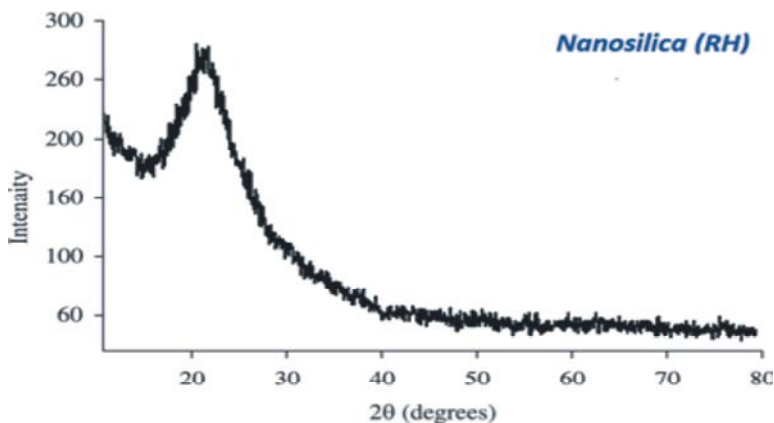
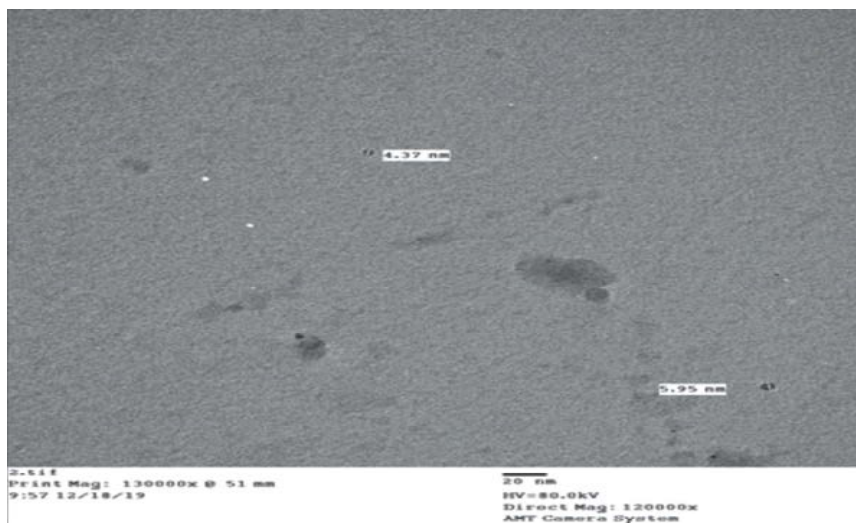


Fig. 2: XRD pattern of Nano-silica extracted from rice husk.



**Fig. 3: TEM micrograph of Nano-silica extracted from rice husk.**

**Field Trial: Yield components of the rice crop as a response to applied Nano-silica and calcium humate under salinity stress.**

Data presented in Table 5 show the effect NSi and CaH, both of them applied at different concentrations, either alone or in combination, on the biological, straw and grain yield of the rice crop under salt stress.

Results show an increase in the rice yield components under the effect of NSi; this increase in mean values of biological, straw and grain yield reached 32, 35 and 26%, respectively. The apparent effectiveness of NSi in enhancing the crop characteristics discussed here may be related to enhance physiological functions and its advantageous impact on the amount of Na in the soil, which lessens the negative effects of salt stress. Furthermore, the results reveal that a high concentration of NSi<sub>3</sub> has a positive effect on rice crop biological, straw and grain yield when compared to either a control or another NSi concentration. This finding corresponds with the findings of [47], who observed that the rice plant expanded as a result of NSi treatment. Other researchers discovered that applying Si to rice plants increased plant height and the number of tillers per m<sup>2</sup> [48], as well as the leaf area index and dry matter production per hill [49]. Previously, [50] demonstrated that the use of silica can increase the leaf angle's uprightness. This can make the leaves more effective at absorbing sunlight, resulting in an increase in photosynthesis and an increase in plant biomass and agricultural seed output. [51] recently demonstrated that NSi particles have an effect on plant physiological properties due to their ability to enter the plant and affect its metabolic processes. Also, [52] reported that the Nano-size of silica allows it to penetrate

the leaf tissue, causing changes in the physicochemical reactions in the cell and activating growth, thereby reducing the adverse effects of saline conditions. These findings may be due to NSi affecting protein synthesis, amino acid synthesis, nutrient uptake and antioxidant enzyme activity.

With regard to the application of CaH results shown in Table 5, all growth parameters of the rice crop were increased by CaH addition, with the mean values of biological, straw and grain yield reaching 7649, 4419 and 3229 kg fed<sup>-1</sup>, respectively, as compared to 5285, 3108, 2177 kg fed<sup>-1</sup> for the control treatment, respectively. This increase in biological, straw and grain yield represents 45, 42 and 48%, respectively; in rice crop grow under salinity stress. The present findings agree with those of [53, 54], who observed that the primary cause of the increase in rice plant productivity was likely the action of CaH, which has a functional group with humic acids, altering the pattern of carbohydrate metabolism and causing an accumulation of soluble sugars that raise the intracellular osmotic pressure, thereby strengthening the plant's resistance against osmotic stresses. Moreover, humic acids boost the availability of essential nutrients for plants' vegetative growth, including potassium, phosphorus and nitrogen. Previously, [55] found that humic acids improved the soil's chemical, physical and biological qualities, minimizing the effect of toxic components and boosting plant resilience to saline stressors, hence influencing the aforementioned traits. Ca also helps in the metabolic process of another nutrient intake, promotes appropriate plant cell elongation, strengthens cell walls and participates in an enzymatic and hormonal process [56].

Table 5: Effect of Nano-silica, calcium humate and their combination on rice crop productivity under salinity stress conditions

Treatments	Conc.	Biological yield (Kg Fed <sup>-1</sup> )	Straw yield (Kg Fed <sup>-1</sup> )	Grain yield (Kg Fed <sup>-1</sup> )
Control	0	5285	3108	2177
Nano-silica (NSi)	NSi <sub>1</sub>	6398	3899	2499
	NSi <sub>2</sub>	6804	3976	2828
	NSi <sub>3</sub>	7651	4732	2919
Mean		6951	4202	2749
Calcium humate (CaH)	CaH <sub>1</sub>	7175	4116	3059
	CaH <sub>2</sub>	7378	4172	3206
	CaH <sub>3</sub>	8393	4970	3423
Mean		7649	4419	3229
NSi + CaH	NSi <sub>3</sub> + CaH <sub>1</sub>	5852	3430	2422
	NSi <sub>2</sub> + CaH <sub>2</sub>	7392	4515	2877
	NSi <sub>1</sub> + CaH <sub>3</sub>	6160	3668	2492
Mean		6468	3871	2597
Mean of conc.	C <sub>1</sub>	6475	3815	2660
	C <sub>2</sub>	7191	4221	2970
	C <sub>3</sub>	7401	4457	2944
LSD at 0.05				
Treatment		348.3	312.4	146.6
Conc.		821.5	640.8	193.0
Treatment* Conc.		1423	1110	334.3

Furthermore, Table 5 shows that, when compared to the control treatment, the application of NSi combined with CaH as a composite result in a beneficial trend in the rice crop component (biological, straw and grain yield). Additionally, NSi<sub>2</sub>+CaH<sub>2</sub> composite application at the medium rate produced the highest yield when compared to other composite rates or control. However, the results obtained indicated that the individual management of either NSi or CaH was more beneficial than the combination application. Such outcomes could result from interference between their respective roles in the plant's mechanisms, i.e., one inhibiting the other's ability to operate. This finding is consistent with that of [57], who found that the concentration of each individual Si application had a favorable impact on rice harvest, grain yield and biological yield.

**Total content of macronutrients and silicon in the rice crop as a response to applied of Nano-silica and calcium humate under salinity stress.**

Results presented in Table 6 show that the macronutrient total content of N, P and K, along with Si, in the straw and grain of the rice crop were significantly increased with all treatments applied as compared to the control. In fact, the growth of rice crops in salt-affected soil considerably decreased the uptake of N, P and K and their accumulation in rice crops (straw and grains). Nevertheless, the harmful impacts of salinity were considerably mitigated when rice plants were treated with both NSi and CaH along with their combination.

When comparing the NSi treatments to the control treatment, the mean value of the macronutrient total content (N, P, K and Si) in the rice crop increased to 23.6, 99.3, 58.5 and 242.9% for straw and similarly increased by 74.9, 83.9, 65.7 and 67.4% in grain, respectively. In addition, when compared to the control, the total amount of N, P, K and Si increased by 27.9, 124.0, 68.0 and 78.4% in straw and by 78.8, 87.2, 75.9 and 27.8% in grain with the CaH treatment. In the combination treatment, total content increased by 9.47, 92.5, 31.9 and 170.4% in straw and by 63.0, 64.8, 63.0, 64.8, 62.4 and 57.1% in grain when compared to the control.

Additionally, the data obtained and displayed in Table 6 indicated a positive trend with the application concentration of either NSi or CaH increased. The overall content of macronutrients grew in tandem with the rising rate of application. However, in composite application, this pattern was different, with NSi<sub>2</sub>+CaH<sub>2</sub> concentration being the superior therapy. These findings are consistent with those of (58), who found that adding NSi particles to saline soil increased Si absorption, increased plant absorption of N, P, K and Si, decreased plant Na uptake, increased plant antioxidant levels and improved photosynthesis efficiency. NSi also helps plants tolerate salt stress by lowering plant turgor pressure and reducing Na uptake. Recently, these results were supported by (59) who demonstrated that the positive effect of silicon could be mediated by a decrease in the uptake and transport of Na<sup>+</sup> and increased uptake and transport of K<sup>+</sup> from roots

**Table 6: Impact of Nano-silica, calcium humate and their combination on macronutrients and silicon total content of rice crop under salinity conditions.**

Treatments	Conc.	N content (Kg fed <sup>-1</sup> )		P content (Kg fed <sup>-1</sup> )		K content (Kg fed <sup>-1</sup> )		Si content (Kg fed <sup>-1</sup> )	
		Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain
Control	0	21.1	19.9	7.15	9.76	24.44	4.81	6.02	19.12
Nano-silica (NSi)	NSi <sub>1</sub>	23.2	28.0	10.67	12.17	31.63	7.11	13.23	26.30
	NSi <sub>2</sub>	25.3	37.5	11.96	18.88	36.51	7.77	16.16	31.32
	NSi <sub>3</sub>	31.9	39.0	20.11	22.81	50.07	9.02	32.53	38.40
Mean	26.1	34.8	14.25	17.95	38.74	7.97	20.64	32.01	
Calcium humate (H)	CaH <sub>1</sub>	21.5	32.1	13.77	15.95	28.86	6.63	8.98	19.99
	CaH <sub>2</sub>	24.0	34.1	14.78	18.17	33.27	7.42	12.51	24.35
	CaH <sub>3</sub>	35.5	40.5	19.54	20.68	61.08	11.33	23.17	28.98
Mean		27.0	35.6	16.03	18.27	41.07	8.46	10.74	24.44
NSi+CaH	NSi <sub>3</sub> + CaH <sub>1</sub>	22.0	26.8	10.01	11.17	27.36	5.96	7.50	25.59
	NSi <sub>2</sub> + CaH <sub>2</sub>	32.1	41.4	18.69	22.89	39.53	10.27	29.17	34.62
	NSi <sub>1</sub> + CaH <sub>3</sub>	24.2	29.1	12.63	14.18	29.79	7.19	12.19	29.90
Mean		23.1	32.4	13.77	16.08	32.23	7.81	16.28	30.04
Mean of Conc.	C <sub>1</sub>	22.2	29.0	11.48	13.09	29.28	6.57	9.90	23.96
	C <sub>2</sub>	27.1	37.7	15.14	19.98	36.44	8.49	19.28	30.10
	C <sub>3</sub>	30.5	36.2	17.43	19.22	46.98	9.18	22.63	32.43
LSD at 0.05									
Treatment		4.25	2.21	3.19	2.60	4.84	1.34	5.18	5.59
Conc.		4.90	3.70	3.69	3.00	5.64	1.76	5.57	5.01
Treatment* Conc.		8.50	6.42	6.39	5.20	9.77	3.05	9.65	8.68

to shoots under salt stress. Silicon seems to affect the acquisition of other essential nutrients such as nitrogen, phosphorus, calcium and other micronutrients as well, thereby improving the growth of plants and, generally, their tolerance to salt stress. Moreover, P concentration and total P contents were increased by adding silicon under saline conditions. The possible causes for this may be associated with both Si-stimulated root activities shown by root dehydrogenase activity and Si-improved P bioavailability in soils due to the chemical competition between phosphate and silicate anions for the sorption sites [60]. Furthermore, the results in Table 6 show that CaH was more efficient than NSi for N, P and K content, but NSi was more successful for Si content in rice plants when compared to the control under the salt stress effect. This may be linked to the favorable effect of CaH on plant growth, which boosted root growth and increased plant absorption of macronutrients. Moreover, CaH contains these nutrients and provides them to the plant.

**Ionic Status in Rice Plant:** In fact, the most important components for a plant to survive salt stress are potassium (K), calcium (Ca) and sodium (Na). Salt tolerance is directly connected with K concentrations because K participates in osmotic regulation and competes with Na ions. Plant salt tolerance needs adaptation to Na<sup>+</sup> toxicity in addition to achieving abundant K<sup>+</sup>, which the plant cell's ability to absorb is

influenced by high external Na<sup>+</sup> concentrations. Thus, to mitigate the detrimental effects of salt stress, we employed NSi, CaH and their composite in rice plants grown in saline soil to inhibit Na absorption and increase K mobility within the plant, resulting in a lower Na/K or Na/Ca ratio. Fig. 4 showed that the application of NSi, CaH and their combination decreased both the Na/K and Na/Ca ratios in the straw and grain of rice crop; the highest reduction was observed at NSi<sub>3</sub> and CaH<sub>3</sub> while the lowest one at NSi<sub>1</sub> and CaH<sub>1</sub>.

Furthermore, the composite treatment was more effective than applying NSi and/or CaH separately; the medium concentration was more effective. Based on these findings, it can be concluded that silicon application reduces the negative effects of salinity by preventing Na<sup>+</sup> uptake by the roots and subsequent movement to the shoots, as well as increasing some beneficial nutrient uptake such as K and Si by increasing the activity of some enzymes, which increased the absorption and transport of K in the plants, thereby improving salinity tolerance in the plants.

The obtained results correspond with the findings of [61], who indicated that K<sup>+</sup> absorption and transport are active processes coupled with an ATP-driven H<sup>+</sup> pump in plasma membranes. The activation of H<sup>+</sup>-ATPase in the membranes is one proposed mechanism for the stimulating effect of Si on K<sup>+</sup> uptake by plants under salt stress, which was corroborated by enhanced H<sup>+</sup>-ATPase



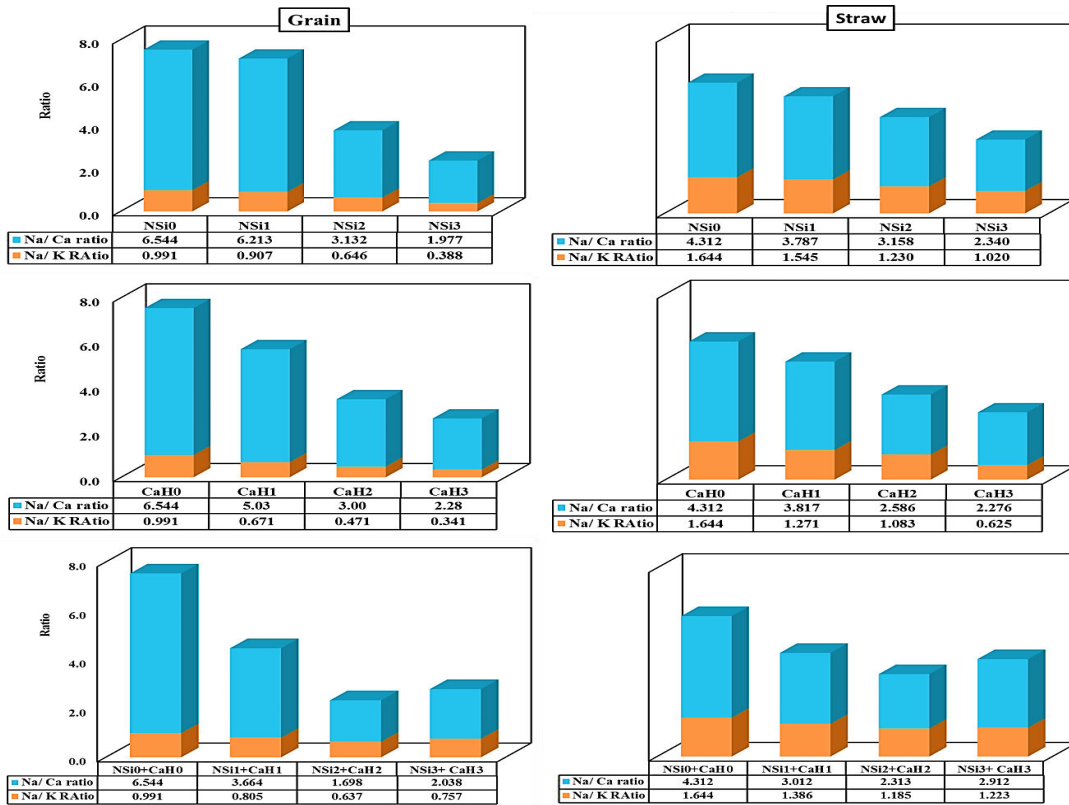


Fig. 4: Effect of Nano-silica, calcium humate and their combination on Na/Ca and Na/K ratios in grain and straw of the rice crop under salinity conditions

activity in salt-stressed plants in the presence of Si. As previously stated, Na<sup>+</sup> is passively taken up by plants and the rate of uptake is mostly influenced by transpiration rate. The lower Na<sup>+</sup> uptake by plants in this study can be explained, at least in part, by silicon's inhibitory influence on transpiration rate.

Recently, [62] observed that applying Si reduced plant total Na accumulation content by decreasing the net Na uptake rate, which could be attributed to the apoplastic blocking impact of Si on Na transport. In this regard, according to [47], foliar application of diverse sources and levels of NSi considerably decreased sodium (Na<sup>+</sup>) content in the plant leaf but dramatically raised potassium (K<sup>+</sup>), K/Na ratio and Si contents when compared to the control treatment.

**Correlation Analysis among Silicon Concentration, Na/K and Na/Ca Ratios in Rice Crop under Salinity Stress:** To make the picture clear, the obtained results as a linear correlation between Si concentration in both straw and grain and Na/Ca or Na/K ratios ( $p < 0.001$  and  $p < 0.01$ ) are shown in Fig. 5. In this study, a significant positive linear correlation was found between Si and

Na/Ca ( $R^2 = 0.959, 0.671$  and  $0.984$ ) in the grain of the rice crop, as well as ( $R^2 = 0.73, 0.927$  and  $0.983$ ) for straw under NSi, CaH and its composite NSi+CaH treatment, respectively. The same trend was observed between NSi and Na/K ratios ( $R^2 = 0.996, 0.878$  and  $0.555$ ) for grain and  $R^2 = 0.999, 0.801$  and  $0.673$  for straw under the same treatments, respectively. Such correlation results indicated that by increasing treatments applied (NSi, CaH and NSi+CaH), Na concentration in rice plants decreased, consequently the Na/Ca or Na/K ratio also decreased. This may be due to the blockage effect of Si on Na transport through the apoplastic pathway [62].

**Chemical Properties of the Experimental Soil:** Table 7 represent the changes in several soil chemical properties that occur when different concentrations of NSi and CaH are applied, either separately or in combination, in saline soil conditions. Results in Table 7 shows that, as compared to a control treatment, the evaluated treatments improved soil pH under salinity stress. This was especially true when different amounts of CaH were administered, either alone or in conjunction with NSi. Obtained data agreement with [63], who stated that the

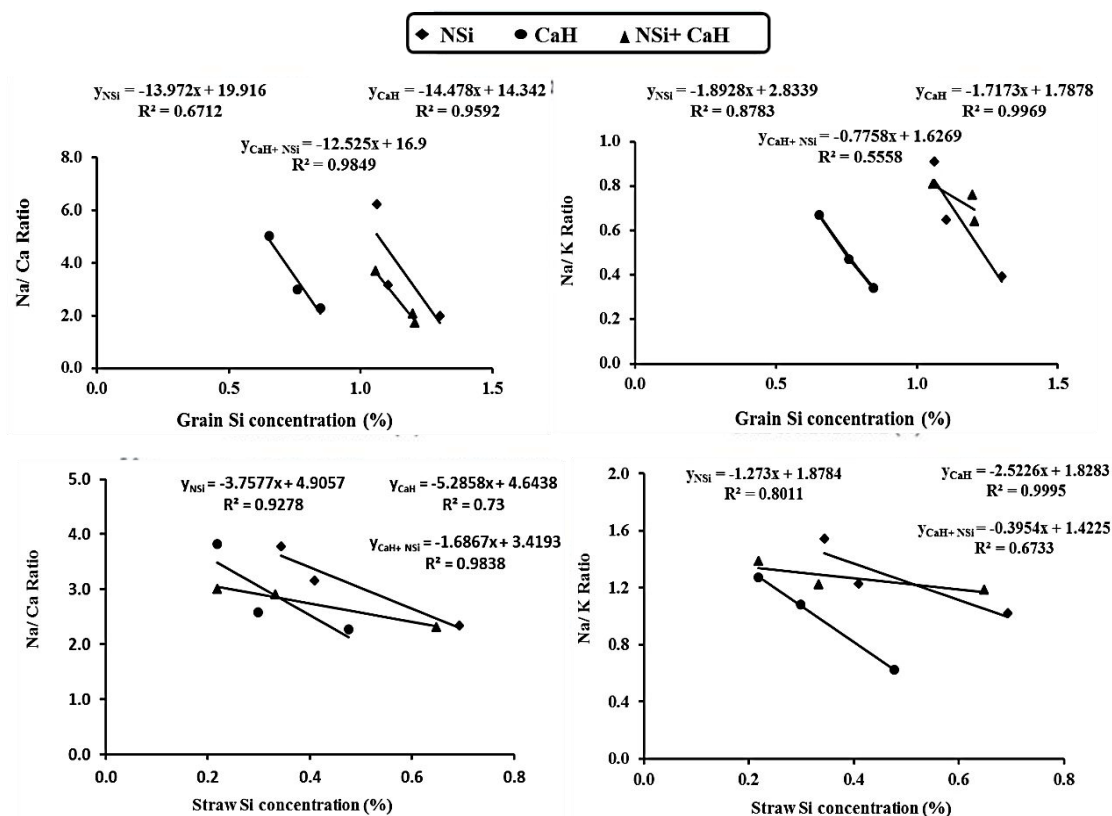


Fig. 5: Correlation coefficient between Si concentrations and Na/Ca and Na/K ratios in the grain and straw of a rice crop under salinity stress

Table 7: Impacts of Nano-silica, calcium humate and their combination on some chemical properties in soil after rice crop harvesting under saline soil condition.

Treatment	Conc.	pH	O.M (%)	EC (dSm <sup>-1</sup> )	SP	SAR	ESP	Anions (meq L <sup>-1</sup> )				Cations (meq L <sup>-1</sup> )			
								CO <sub>3</sub> <sup>2-</sup>	HCO <sub>3</sub> <sup>-</sup>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Na <sup>+</sup>	K <sup>+</sup>
Control		8.02	1.01	9.93	81	12.2	15.6	-	11.56	58.3	47.5	24.4	27.67	64.0	1.40
Nano-silica (NSi)	NSi <sub>1</sub>	8.02	1.02	8.00	81	12.9	15.3	-	10.80	50.0	42.04	17.93	24.50	59.2	1.21
	NSi <sub>2</sub>	8.01	1.03	7.95	79	11.0	13.3	-	8.64	49.2	37.25	17.80	25.00	51.0	1.29
	NSi <sub>3</sub>	8.02	1.05	7.89	79	10.6	12.8	-	7.56	45.3	41.22	18.18	25.23	49.2	1.42
Mean		8.02	1.03	7.95	79	11.5	13.8	-	9.00	48.1	40.17	17.97	24.91	53.16	1.31
Calcium humate (CaH)	CaH <sub>1</sub>	7.84	1.15	7.84	85	12.2	14.6	-	4.00	40.0	49.7	15.30	23.62	53.7	1.10
	CaH <sub>2</sub>	7.77	1.25	7.73	86	10.8	13.1	-	4.32	43.5	44.1	17.41	24.15	49.2	1.13
	CaH <sub>3</sub>	7.71	1.42	7.60	85	9.7	11.8	-	5.40	41.6	43.2	19.50	24.22	45.3	1.20
Mean		7.77	1.27	7.72	85	10.9	13.1	-	4.57	41.7	45.7	17.40	24.00	49.4	1.14
Nsi + CaH	NSi <sub>1</sub> +CaH <sub>1</sub>	7.99	1.10	8.70	82	12.2	14.6	-	9.72	60.0	53.67	27.50	29.41	65.2	1.28
	NSi <sub>2</sub> +CaH <sub>2</sub>	7.98	1.20	8.30	81	10.5	12.7	-	7.02	51.0	58.00	28.50	29.50	56.5	1.44
	NSi <sub>3</sub> +CaH <sub>3</sub>	7.95	1.30	8.56	81	10.9	13.1	-	6.48	65.5	52.47	29.98	32.41	60.6	1.43
Mean		7.98	1.20	8.52	81	11.2	13.5	-	7.74	58.8	54.71	28.66	30.44	60.8	1.38

decrease in soil pH reflects the effect of calcium ions reacting with bicarbonate to precipitate as calcite and then protons (H<sup>+</sup>) are released in soil solution, neutralizing the hydroxide ions (OH<sup>-</sup>) and thus decreasing soil pH. Furthermore, [64] elucidate that there is a negative correlation between soil pH and HA. This is because HA addition can regulate excess Na, reducing its toxic and osmotic effects and raising the availability of nutrients for plant growth.

The results demonstrated that NSi treatments had only minor effects on SP. Under salinity stress, however, there was no difference in SP after utilizing NSi in conjunction with CaH. Although [65] showed that applying NSi to the soil enhanced the saturation percentage, despite this, there is a little rise in SP content when different CaH concentrations are utilized as compared to control treatment.

Results regarding organic matter (OM) indicate a pattern that is contrary to that of pH, with OM content increasing in response to applications of CaH, either alone or in conjunction with treatments involving NSi, compared to the control treatment. While applying NSi alone had little effect on organic matter in saline soil, comparable evidence supports the findings of [66], who discovered that CaH functions as organic matter to support soil microbes. This increase could be attributed to the production of organic linkers, which improve soil consistency by improving porosity and particle adhesion strength, lowering water consumption for soil moisture maintenance during watering and boosting soil water saturation [67].

Furthermore, all treatments had lower soil electric conductivity (EC) than the control treatment (Table 7). Furthermore, NSi, CaH and their combination treatments contributed to EC reductions of 23.5%, 20.5% and 16.4%, respectively, as compared to the control. The findings are consistent with those of [65], who discovered a strong negative association between soil soluble salt and NSi content. According to [68], the favorable effect of additional Ca sources on reducing EC in the soil may be ascribed to the leaching of Na<sup>+</sup> from the soil profile caused by Ca<sup>++</sup> substitution. With rising rates, the interaction between calcium supplies and rates on saline soil was significantly reduced. HA and soil EC have a negative correlation because HA can reduce excess Na, which

lessens its harmful and ionic effects. This lowers soil EC and increases plant nutrient availability [64].

**Soluble Cations, Anions and Soil sodicity:** Table 7 presents the findings of an investigation into the effects of NSi, CaH, and NSi combined with CaH on soluble cations and anions in soil. The findings showed that, as compared to the control, all soluble anions (HCO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup>, and SO<sub>4</sub><sup>-</sup>) and cations (Ca<sup>++</sup>, Mg<sup>++</sup>, K<sup>+</sup>, and Na<sup>+</sup>) were typically reduced with all treatments administered. The results show that while Si is responsible for plant adaptation to salt stress, CaH is more effective at moving soil elements. The highest reduction was observed with calcium humate, followed by NSi+CaH and NSi.

The exchangeable sodium percentage (ESP) and sodium absorption ratio (SAR) calculations can also be used to express salinity. The soil sodicity values, SAR, and ESP are also shown in Table 7. When all treatments were applied, the values were lower than for the control. As the quantities of NSi and CaH increased, SAR and ESP decreased. When CaH and NSi were mixed, the SAR ranged from 10.5 (NSi<sub>2</sub>+CaH<sub>2</sub>) to 12.2 (NSi<sub>3</sub>+CaH<sub>1</sub>) treatment, as well the ESP ranged between 12.7 (NSi<sub>2</sub>+CaH<sub>2</sub>) and 14.6 (NSi<sub>3</sub>+CaH<sub>1</sub>). Additionally, when 4% CaH was used instead of other treatments, SAR and ESP decreased more. In comparison to other treatments, [65] discovered that the application of NSi produced the largest SAR, with average increases of 20%. SAR behaves in the same way as sodium. SAR data demonstrated the significant amount of excluded or underutilized sodium in the soil as a result of silicon's beneficial influence on the root absorption mechanism.

Table 8: Effect of applied Nano-silica with or without calcium humate applications on the macronutrients and silicon availability in the soil after a rice crop harvested under saline soil conditions.

Treatment	Conc.	Available nutrients (mg kg <sup>-1</sup> )			
		N	P	K	Si
Control 0		113	4.10	315	204
Nano- silica (NSi)	NSi <sub>1</sub>	109	6.00	314	241
	NSi <sub>2</sub>	121	7.03	316	269
	NSi <sub>3</sub>	122	8.45	349	380
Mean	117	7.16	326	296	
Calcium humate (CaH)	CaH <sub>1</sub>	113	4.63	342	238
	CaH <sub>2</sub>	118	6.47	368	243
	CaH <sub>3</sub>	144	8.80	384	252
Mean		115	6.63	365	245
NSi+CaH	NSi <sub>3</sub> +CaH <sub>1</sub>	113	4.53	290	228
	NSi <sub>2</sub> +CaH <sub>2</sub>	120	8.77	342	265
	NSi <sub>1</sub> +CaH <sub>3</sub>	116	7.67	334	234
Mean		116	6.99	322	242
Mean of conc.	C <sub>1</sub>	112	5.05	316	235
	C <sub>2</sub>	120	7.50	342	259
	C <sub>3</sub>	127	8.31	355	289
<b>LSD 0.05</b>					
Treatment		11.29	1.03	43.7	10.9
Conc.		11.66	1.12	30.3	9.67
Treatment*Conc.		20.19	1.92	52.5	16.8

### Macronutrients and Silicon Availability in the Soil after the Rice Crop Is Harvested:

The availability of macronutrients and silicon in soil following rice crop harvesting was evaluated, and the results are shown in Table 8. Data obtained show that NPK and silicon availability were generally increased for all investigated treatments as compared to controls. Furthermore, using a high dosage of both NSi and CaH separately boosted nutrient availability in soil when compared to other formulations. Also, the results in Table (8) demonstrate that adding C<sub>3</sub> treatments raised soil availability of N, P, K, and Si by about 12.4, 102.7, 12.7, and 41.7%, respectively. Compared to other combinations of soil concentrations, the NSi<sub>2</sub>+CaH<sub>2</sub> treatment recorded greater values of N, P, K, and Si when NSi was mixed with CaH. This could be attributable the use of NSi boosted plant health, which raised root secretions and nutrient solubility. Furthermore, CaH application resulted in a decrease in soil pH and EC, which was reflected in nutrient availability. This finding is consistent with (64), who state that the addition of HA can regulate excess Na, reducing toxic and osmotic effects, lowering soil pH and EC, and increasing nutrient availability for the plant.

### CONCLUSION

Recycling waste, such as rice husk and waste from plants, to create new compounds (Nano-silica and humic materials) that can boost output, enhance soil fertility and aid in plant resistance to climate change is the most significant issue affecting the agriculture sector. Based on the earlier findings, it can be deduced that under salinity stress, applied of Nano-silica and calcium humate as organic materials, either separately or in combination at varying rates, enhanced rice straw and grain yield along with nutrient total content (N, P, K and Si) and lowered Na total content. While all applied treatments had a significant impact on soil chemical properties such as pH and EC, boosting OM and accessible NPK soil content also improved soil production under saline conditions.

### ACKNOWLEDGMENT

The paper's authors acknowledge and thank the Development of Soil Conditioners Project, Dept. of Physics and Chemistry of Soil, Soils, Water and Environ. Res. Inst., Agric. Res. Centre (ARC), Giza, Egypt, for providing all of the tools and resources needed for carrying out this research.

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