



Treatment of soil with nanosilica particles derived from rice husk: influence of substrate ageing on soil resistance to detachment

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Abstract: No studies have explored how much ageing of rice husk, used as substrate for nanosilica production, alters apparent particle size distribution in this substrate, and therefore modifies nanosilica-soil interactions. To fill this gap, this study has investigated the effects of nanosilica derived from fresh and aged rice husk on soil physical properties and particle detachment (D_c) in paddy soils through flume experiments on samples collected in Northern Iran. Nanosilica obtained from aged husk showed an increase in mean apparent particle size by 96% compared to the soil amendment derived from fresh residues. Soil treated with fresh husk nanosilica exhibited significantly higher (+106%) water-stable aggregates and lower (−7.4%) bulk density compared to the control, while the differences from aged husk treatments were +86 % and −6.3%, also in this case significant. Flume experiments revealed a significant reduction (−21%) in D_c following application of nanosilica from fresh rice husk, while no effect was found for the soil amendment derived from the aged biomass. Compared to the control, D_c decreased by 8.1% with aged husk and by approximately 21% with fresh husk. D_c can be accurately estimated by hydraulic predictors (shear stress, stream power, and unit stream power) using a shifted power function ($R^2 = 0.80–0.96$). These models show that, across treatments, the effective hydraulic threshold parameter generally increases from control to amended soils, indicating enhanced resistance to detachment initiation.

Keywords: Soil erosion; Soil structural stability; Agricultural residues; Biomass transformation; Soil resilience.

1 INTRODUCTION

Soil erosion is one of the most severe threats to agricultural sustainability, particularly in intensively managed croplands, where structural degradation reduces resistance to hydraulic forces (Lal, 2001; Pimentel et al., 1995). In paddy systems, erosion processes are governed not only by rainfall impact, but also by overland flow during irrigation (Arunrat et al., 2022; Chen et al., 2013). Repeated tillage and prolonged bare-soil periods weaken aggregate stability and increase soil susceptibility to detachment (Bronick and Lal, 2005; Six et al., 2000b). Since soil detachment is a micro-scale process driven by inter-particle bonding strength and pore-scale hydraulic stresses, effective mitigation strategies must operate at the level of soil structure formation and stabilization (Morgan, 2009; Nearing et al., 1997).

Recent advances in nanotechnology have opened new horizons for enhancing soil physical resilience through targeted modification of particle interactions and surface chemistry (Kah et al., 2018). Among nanomaterials, silica nanoparticles are particularly promising due to their chemical stability, environmental compatibility, and capacity to form linkages with mineral and organic components (Wang et al., 2012). Silica-based nanomaterials have been explored in agriculture primarily for nutrient delivery and plant growth enhancement (Nair et al., 2010; Raliya et al., 2016). However, their potential role in improving soil structural stability and reducing hydraulic erosion is still under-investigated.

Rice husk represents an abundant agricultural residue rich in amorphous silica (Kalapathy et al., 2000; Yalcin and Sevinc, 2001). These characteristics make this residue an attractive and sustainable substrate for nanosilica production (Kalapathy et al., 2000; Wang et al., 2012). Transforming rice husk waste into

functional nanomaterials agrees with circular economy principles and offers an environmentally responsible alternative to synthetic stabilizers. However, the physico-chemical characteristics of nanosilica do not solely depend on the extraction process, but they are strongly influenced by the structural and chemical condition of the raw biomass (Kalapathy et al., 2000; Wang et al., 2012). During storage and ageing, rice husk undergoes physico-chemical changes, including partial degradation of organic components, oxidation, and structural rearrangement of silica elements (Yalcin and Sevinc, 2001). These changes may significantly affect nanoparticle size distribution, surface reactivity, and aggregation behavior after extraction. Through surface bonding, electrostatic interactions, and potential formation of siloxane bridges, nanosilica may increase aggregate stability and reduce soil erodibility (Sorum and Sorum, 2026). Smaller particles typically exhibit higher specific surface area and surface energy, enhancing their ability to interact with clay minerals, organic matter, and micro-aggregates (Kah et al., 2018; Six et al., 2004).

Ageing of rice husk alters particle size distribution in this substrate and therefore modifies nanosilica-soil interactions. Specifically, nanosilica derived from fresh rice husk could exhibit a smaller particle size, higher reactivity, and stronger soil-stabilizing capacity compared to nanosilica derived from aged husk. Despite a large body of literature on the anti-erosive effects of rice husk used as soil amendments (e.g., Li et al., 2025; Parhizkar et al., 2021, 2023; Shen et al., 2021), the role of biomass ageing in controlling nanosilica characteristics - and consequently soil erosion resistance - remains poorly understood. We hypothesized that ageing of rice husk significantly modifies the physico-chemical properties of extracted nanosilica, particularly particle size distribution. Moreover, it can also be

supposed that these changes alter the effectiveness of nanosilica on soil structural stability and resistance to hydraulic detachment.

To verify these hypotheses, we conducted flume experiments - that offer a controlled environment to isolate the influence of defined hydraulic conditions on detachment dynamics under (Zhang et al., 2008) - on samples of soils from paddy fields of Northern Iran, which were treated with fresh and aged rice husk. These samples were subjected to scouring due to the overland flow at a variable soil slope and water discharge. By integrating nanoscale characterization with macroscale erosion measurements, it becomes possible to elucidate the mechanistic pathways through which bio-derived nanomaterials influence soil resistance. The specific objectives of the study were therefore: (i) to quantify differences in nanosilica apparent particle size produced from fresh and aged rice husk; (ii) to evaluate the effects of these materials on soil bulk density, water-stable aggregates, detachment capacity, and hydraulic erosion parameters; and (iii) to investigate how soil detachment capacity is influenced by important hydraulic erosion parameters - e.g., soil erodibility, and critical values of shear stress and stream power (Nearing et al., 1997; Knappen et al., 2007) - linking soil structural properties to flow-induced mechanical stresses. By explicitly linking biomass condition, nanoparticle characteristics, and soil erosion mechanics, this research provides a novel contribution to the development of nano-enabled strategies for sustainable soil management in intensively cultivated paddy systems.

2 MATERIAL AND METHODS

2.1 Study area

Paddy fields in the Rice Research Institute of Iran (Rasht city, Guilan Province, Northern Iran, 37°12'18" N; 49°38'28" E, elevation of 7 m above the mean sea level) were chosen as the study area (Figure 1). The climate is typically Mediterranean, Csa type according to the Köppen-Geiger classification (Kottek et al., 2006). The average annual temperature and rainfall are 16.3 °C and 1360 mm, respectively (National Meteorological Institute of the Islamic Republic of Iran).

These paddies located are intensively managed and subjected to continuous rice cultivation, and chemical fertilizers are frequently applied to maximize yield. In many fields, nitrogen, phosphorus, and potassium fertilizers are often used in excessive amounts. Such long-term fertilization practices, combined with intensive tillage operations, have progressively weakened soil structure and increased the susceptibility of soil particles to detachment. Moreover, rice cultivation in the region is limited to the first half of the year, and during the remaining six months, the fields are commonly left fallow with minimal surface protection. This prolonged bare-soil period exposes the soil to rainfall and overland flow, thereby enhancing soil aggregate breaking and detachment processes. These conditions make the study area particularly suitable for evaluating factors influencing soil detachment capacity (Parhizkar et al., 2025).

Furthermore, the presence of multiple rice cultivars, including *Hashemi*, *Ali Kazemi*, *Kadus*, and *Domsiah*, results in the continuous and abundant production of rice husk residues across the region. Large quantities of rice husk are either freshly generated after harvest or stored for extended periods, leading to the natural availability of both fresh and aged rice husk materials. This unique situation provides an ideal opportunity to investigate and compare the effects of nanosilica derived from fresh and aged rice husk on soil detachment behavior, thereby strengthening the relevance of the selected study area to the objectives of the present research.

2.2 Production of silica nanoparticles

In this study, silica nanoparticles were synthesized from rice husk based on the method described by Wang et al. (2012). Initially, raw rice husk was treated by refluxing in a 10 wt% hydrochloric acid solution for two hours under ambient pressure to remove inorganic and organic contaminants. The treated husk was then thoroughly washed with deionized water until a neutral pH was obtained, followed by oven drying at 100 °C for 24 hours. The dried material was subsequently subjected to thermal treatment in a muffle furnace at 700 °C for two hours, producing

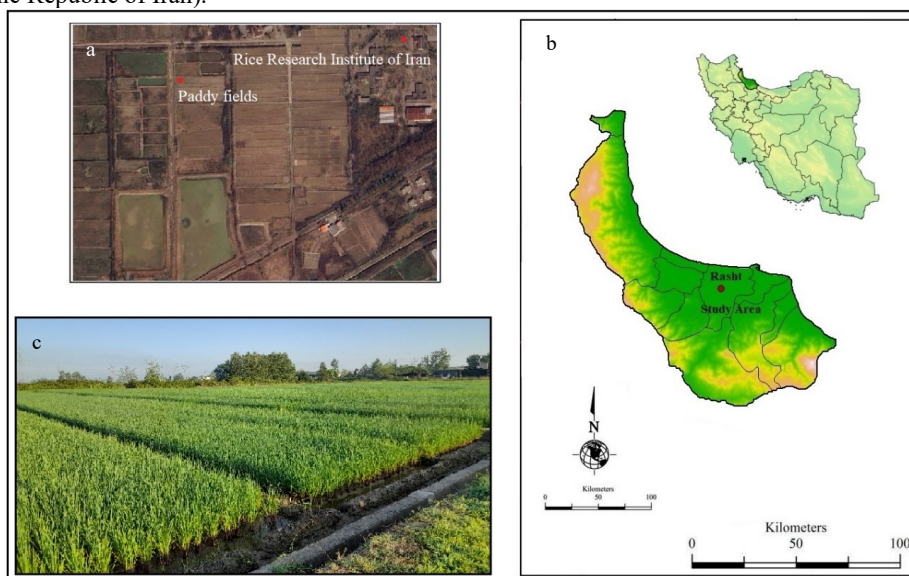


Fig. 1. Location of the study area in Rasht (Guilan Province, Northern Iran). The figure shows (a) an aerial view (Source: Google® Map®) of representative paddy fields where soil sampling and experimental treatments were conducted, (b) the geographical position of the study area within Iran and Guilan Province, and (c) a ground-level photograph of the experimental fields at the Rice Research Institute of Iran.

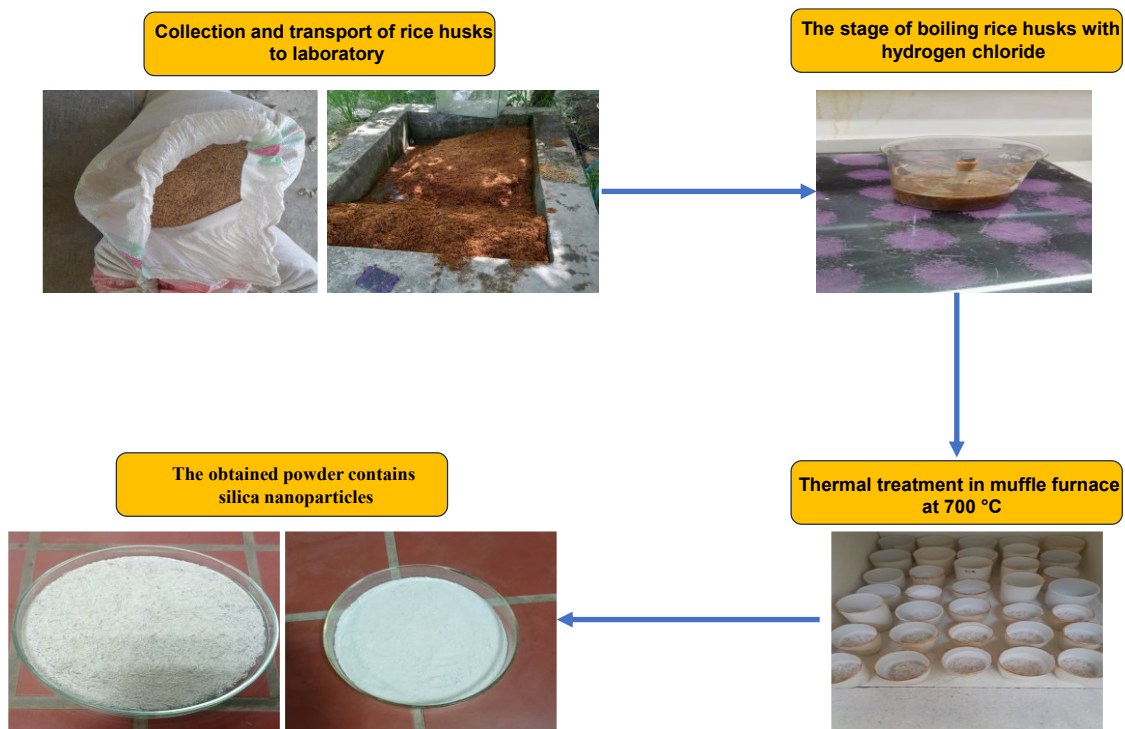


Fig. 2. Schematic representation of the production process of nanosilica from rice husk, including acid pre-treatment, washing, drying, and thermal treatment in a muffle furnace leading to the formation of silica nanoparticles.

a fine and white nanosilica powder (Figure 2). Particle size was estimated from FESEM images by measuring the diameter of randomly selected particles ($n > 20$) using image analysis software (Fig. 1SM). The reported values represent apparent particle size and may reflect particle aggregation. The chemical and structural properties of the synthesized nanosilica were characterized using Fourier-Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD) (Fig. 2SM and Table 1SM). FTIR analysis was used to identify functional groups and confirm the presence of silica-related bonds, while XRD analysis provided information on the structural organization and crystallinity of the material. It should be noted that these techniques do not provide direct measurements of particle size distribution. Therefore, the reported differences in particle size between treatments should be interpreted as indicative of variations in aggregation behavior and structural arrangement rather than precise nanoparticle size measurements.

After synthesis, silica nanoparticles were applied to the paddy fields. The application rate was 2% by weight, and the incubation time lasted seven months, spanning from land preparation to harvest. The rate and incubation time were selected based on a previous study of Parhizkar (2025), who found this time to be an effective duration for evaluating the influence of silica nanoparticles on soil properties.

The incubation period was conducted under field conditions, and, therefore, environmental variability (e.g., temperature, moisture fluctuations) reflects realistic agricultural conditions rather than controlled laboratory settings.

2.3 Description of flume experiments

Following the incubation period, soil detachment capacity was assessed using soil samples randomly collected within each treatment plot from the experimental paddy fields (treated with rice husk-derived nanosilica or untreated, the latter samples considered as control) to minimize spatial bias. Samples were extracted from the upper 0–20 cm layer using a steel ring with a

diameter of 0.1 m and a height of 0.05 m (Zhang et al., 2008).

Scouring experiments to measure soil detachment capacity were performed in a rectangular hydraulic flume with an adjustable slope (3 m in length and 0.2 m in width) (Figure 3).

A circular opening (0.1 m in diameter) was installed near the downstream section of the flume bed to accommodate soil samples and facilitate the measurement of soil particle detachment under overland flow. Before each experimental run, soil surfaces were uniformly pre-wetted by gentle spraying for 24 hours to establish comparable initial moisture content.

During the experiments, hydraulic parameters were carefully recorded. Water discharge (Q , L/s) was measured in five replicates using a graduated cylinder. Flow depth (h , mm) was measured with a digital level probe accurate to ± 1 mm over the sample with three measurements for each run. Mean velocity (V , m/s) was calculated as the ratio between Q and the flow cross-section area ($0.2 \text{ m} \times h$) after applying correction coefficients proposed by Abrahams et al. (1986) to account for the hydraulic regime (laminar, transitional or turbulent).

Each test was terminated when the depth of soil removal within the sampling ring reached approximately 0.015 m or when the maximum experimental duration of five minutes was achieved. In order to reduce the influence of uneven detachment within the sample, the experiment ended when the eroded soil depth was 0.015 m or after five minutes, in accordance with previous studies (e.g., Nearing et al., 1991; Zhang et al., 2008). Detached sediments were collected and oven-dried at 105 °C for 24 hours to determine eroded soil weight.

2.4 Determination of soil physical properties

In addition to soil detachment capacity, additional soil samples were taken from the same paddy fields in points adjacent to previous sampling, to measure water-stable aggregation (WSA), using the wet sieving technique described by Kemper and Rosenau (1986) and bulk density (BD), based on the clod method as outlined by Radcliffe and Simunek (2018).



Fig. 3. Experimental setup of the hydraulic flume used to measure soil detachment capacity (D_c). The system includes a water supply tank, adjustable slope channel (3 m length, 0.2 m width), and a circular sample holder (0.1 m diameter) installed near the downstream section for testing soil samples under controlled overland flow conditions.

2.5 Experimental design

To quantify soil detachment capacity under flowing water, fifteen experimental hydraulic conditions were established by combining three slope gradients (0.65, 1.52 and 2.23%), representative of the longitudinal profile of a paddy field in the area, with five different flow discharge rates (1.5, 2.0, 2.5, 3.0, and 3.5 L/s) (Table 1). Each of the fifteen hydraulic conditions was repeated three times, totaling 135 experimental runs (three soil conditions, i.e., aged and fresh rice husk, and control without any treatment $\times$ three slope gradients $\times$ five flow rates $\times$ three replications).

2.6 Calculation of soil detachment capacity and hydraulic parameters

The soil detachment capacity (D_c , kg/m² s) for each repeat under each combination of water discharge and soil condition was calculated by equation (1):

$$D_c = \frac{\Delta M}{A \Delta t} \quad (1)$$

where ΔM is the dry weight of detached soil (kg), A is the area of the soil sample (m²), and Δt is the experiment duration (s).

The shear stress, according to Nearing et al. (1997), was estimated using the following equation (2):

$$\tau = \rho g R S \quad (2)$$

where ρ is the water density (kg/m³), g is the acceleration of gravity (m/s²), R is the hydraulic radius (m), and S is the bed slope (m/m).

The stream power (ω , kg/s³) was obtained using the following equation:

$$\omega = \rho g R S V = \tau V \quad (3)$$

The unit value of the stream power (ω_u , m/s) was calculated as:

$$\omega_u = S V \quad (4)$$

According to Govers et al. (2007), D_c was modelled from τ , ω and ω_u using three non-linear regression equations:

$$D_c = a(\tau - b)^c \quad (5)$$

$$D_c = a'(\omega - b')^{c'} \quad (6)$$

and

$$D_c = a''(\omega_u - b'')^{c''} \quad (7)$$

where a , a' , a'' , b , b' , b'' , c , c' and c'' are regression coefficients. The slopes of the equations are three soil erodibility coefficients, whereas the intercepts are the critical values of the shear stress, stream power and unit stream power. In other words, the detachment process acts when the difference between the actual τ , ω and ω_u exceeds these critical values. The goodness of fit of these equations was checked using the coefficients of determination (R^2).

2.7 Statistical analysis

Differences in water stable aggregates, bulk density and soil detachment capacity (response variables) among the studied conditions (independent variables with three levels: soil treated with fresh or aged rice husk, and control without treatment) were examined using one-way analysis of variance (ANOVA). Mean pair-wise comparisons were performed using Tukey's test at a significance level of $p < 0.05$. Before statistical analysis, data distribution normality was verified using Shapiro-Wilk's test, while variance homogeneity was assessed via Levene's test.

All statistical analyses - including non-linear regressions - were performed using OriginPro, version 2026, OriginLab Corporation, Northampton, MA, USA.

Table 1. Hydraulic conditions applied in the flume experiments to measure soil detachment capacity (D_c), including slope gradient (S), water discharge (Q), shear stress (τ), stream power (ω), and unit stream power (ω_u). Values represent the range of hydraulic forcing conditions tested across all soil treatments (control, and soils amended with nanosilica derived from fresh and aged rice husk).

Slope (S , %)	Water discharge (Q , L/s)	Shear stress (τ , Pa)	Stream power (ω , kg/s ³)	Unit stream power (ω_u , m/s)
0.65	1.5	0.494	0.441	0.006
	2.0	0.553	0.582	0.007
	2.5	0.652	0.715	0.007
	3.0	0.698	0.851	0.008
	3.5	0.743	0.985	0.009
1.52	1.5	1.001	1.042	0.016
	2.0	1.142	1.375	0.018
	2.5	1.267	1.704	0.020
	3.0	1.488	2.011	0.021
	3.5	1.632	2.321	0.022
2.23	1.5	1.315	1.540	0.026
	2.0	1.449	2.041	0.031
	2.5	1.656	2.525	0.034
	3.0	1.841	3.002	0.036
	3.5	2.059	3.464	0.038

3 RESULTS

3.1 Variability in nanosilica particle size produced from fresh and aged rice husk

A substantial difference in nanosilica particle size was found between fresh and aged rice husk (Table 2). In more detail, the apparent particle size produced from fresh rice husk was 15.3 nm, with a minimum of 8.19 nm and a maximum of 23.2 nm. In contrast, nanosilica derived from aged rice husk showed a much larger mean size (363 nm), and minimum and maximum values ranged from 279 to 504 nm. The standard deviation was considerably higher for aged husk nanosilica compared to fresh husk nanosilica.

Table 2. Apparent particle size characteristics (nm) of nanosilica derived from fresh and aged rice husk, including mean, minimum, maximum, and standard deviation values. These values reflect differences in aggregation state and structural organization inferred from material characterization, rather than direct measurements of primary nanoparticle size.

Rice husk condition	Mean	Minimum	Maximum	Std. Dev.
<i>Fresh</i>	15.3	8.19	23.2	2.96
<i>Aged</i>	363	279	504	52.2

3.2 Variability of bulk density and aggregate stability of soils treated with nanosilica from fresh and aged rice husk

WSA and BD were significantly different among the studied conditions ($p < 0.0001$). In more detail, the mean WSA was significantly ($p < 0.0001$) higher in the soil amended with nanosilica produced from fresh rice husk compared to the treatment with nanosilica of aged rice husk and the control soil (Figure 3).

Table 3. Results of one-way analysis of variance (ANOVA) for soil detachment capacity (D_c , kg/m² s), bulk density (BD , kg/m³), and water-stable aggregates (WSA , %) measured in control soil and soils treated with nanosilica derived from fresh and aged rice husk. Reported values include degrees of freedom (df), sum of squares, mean square, F-value, and significance level (Prob > F). Statistical significance was assessed at $p < 0.05$.

Variable	Degrees of Freedom	Sum of squares	Mean square	F-value	Prob>F
D_c	2	0.014	0.007	5.24	0.009
BD	2	107885	53942	45.312	<0.0001
WSA	2	0.885	0.442	312.036	<0.0001

The values of BD measured in the control (1510 ± 46.1 kg/m³) and soil treated with nanosilica produced from aged rice husk (1492 ± 36 kg/m³) were significantly ($p < 0.0001$) higher compared to the soil treated with nanosilica produced from fresh rice husk (1398 ± 12.1 kg/m³) (Figure 4).

3.3 Variability of particle detachment capacity of soils treated with nanosilica from fresh and aged rice husk

The D_c was significantly different between the control soil and the soils treated with nanosilica from fresh and aged rice husk ($p < 0.01$) (Table 3). More specifically, the control soil showed the highest D_c across the applied water discharge and under all soil slopes (0.197 ± 0.039 kg/m² s), indicating greater susceptibility to erosion. Soils amended with nanosilica from aged rice husk exhibited lower D_c (0.181 ± 0.032 kg/m² s) compared to the control, but this value was higher compared to fresh husk treatments (0.154 ± 0.034 kg/m² s) (Figure 5).

3.4 Estimation of soil erodibility and critical shear stress, stream power and unit stream power for the soils treated with nanosilica from fresh and aged rice husk

Table 4 reports the coefficients of the fitted models $D_c = a(x - b)^c$ for the three hydraulic predictors (shear stress, stream power, and unit stream power, considered as the independent variable x) under the different soil conditions. The models provided good fits to the data (R^2 ranging from 0.80 to 0.96). Across the gradient from control to aged and fresh rice husk treatments, both parameters a and b generally increased (except when the predictor was τ), indicating a progressive shift in the detachment response. In several cases, the parameter b was close to zero or associated with relatively large standard errors, especially under control conditions. This suggests limited sensitivity of the model in identifying a distinct hydraulic threshold. The highest coefficients of determination were obtained when ω_u was used as a predictor, although some parameter estimates under control conditions were characterized by large uncertainties (Table 4 and Figure 6).

4 DISCUSSION

4.1 Influence of rice husk ageing on nanosilica apparent particle size and structural characteristics

The noticeable difference in apparent particle size (+96%) between nanosilica derived from fresh and aged rice husk clearly indicates that biomass ageing plays a fundamental control on silica transformation before extraction. It should be emphasized that particle size differences inferred in this study are indicative

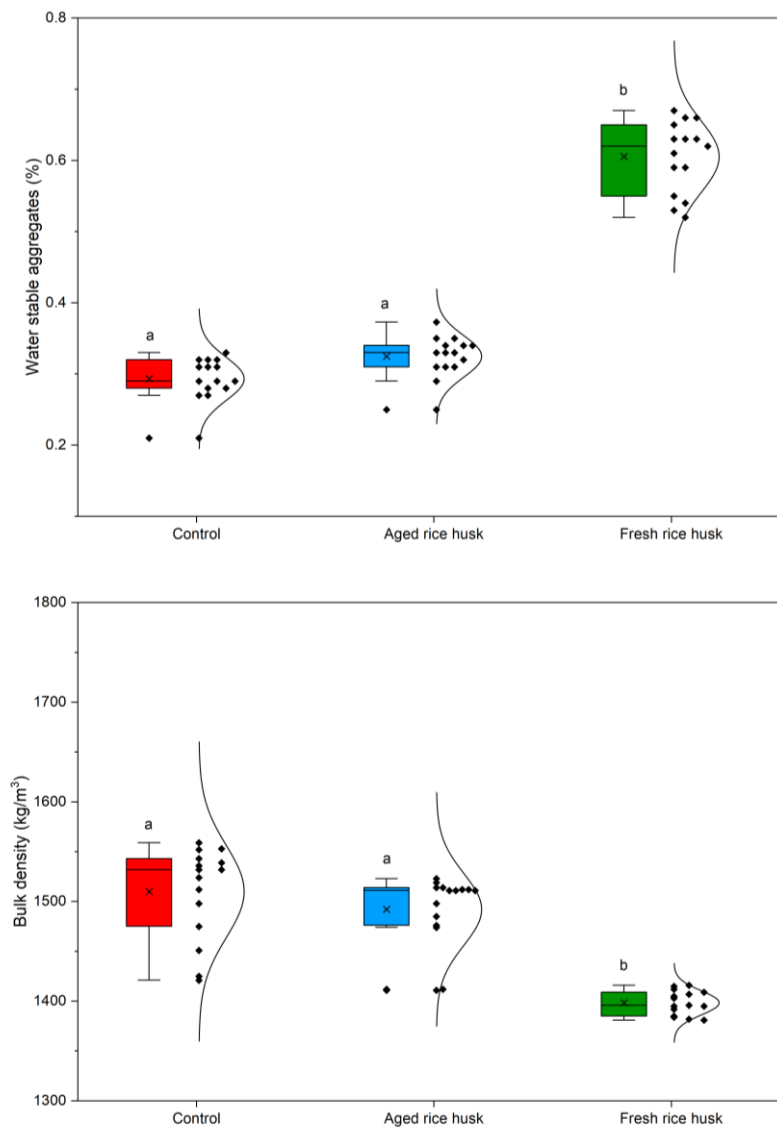


Fig. 4. Mean ($\pm$ standard deviation, $n = 15$) of water stable aggregates and bulk density measured on soil samples treated with nanosilica derived from aged and fresh rice husk compared to the untreated control in paddy fields (Rasht, Guilan Province, Northern Iran). Different letters indicate statistically significant differences among treatments according to Tukey's test ($p < 0.05$).

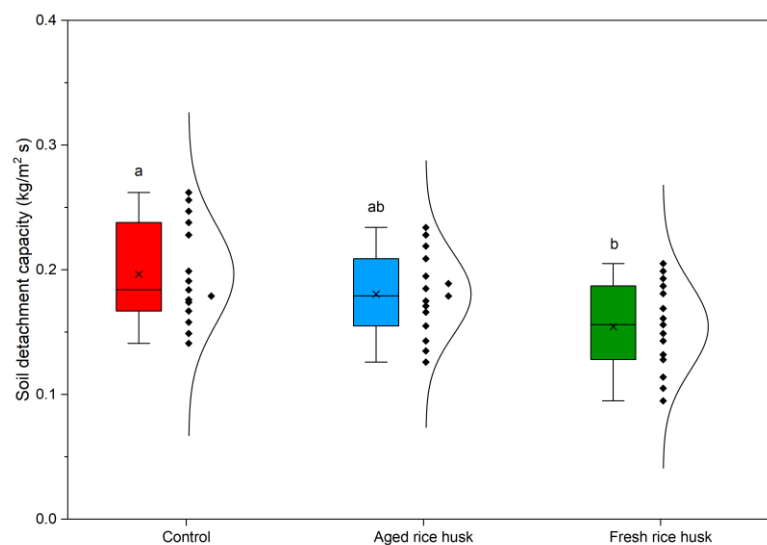


Fig. 5. Mean ($\pm$ standard deviation, $n = 15$) of soil detachment capacity measured on soil samples treated with nanosilica from aged and fresh rice husk compared to the untreated control in paddy fields (Rasht, Guilan Province, Northern Iran). Different letters indicate statistically significant differences among treatments according to Tukey's test ($p < 0.05$).

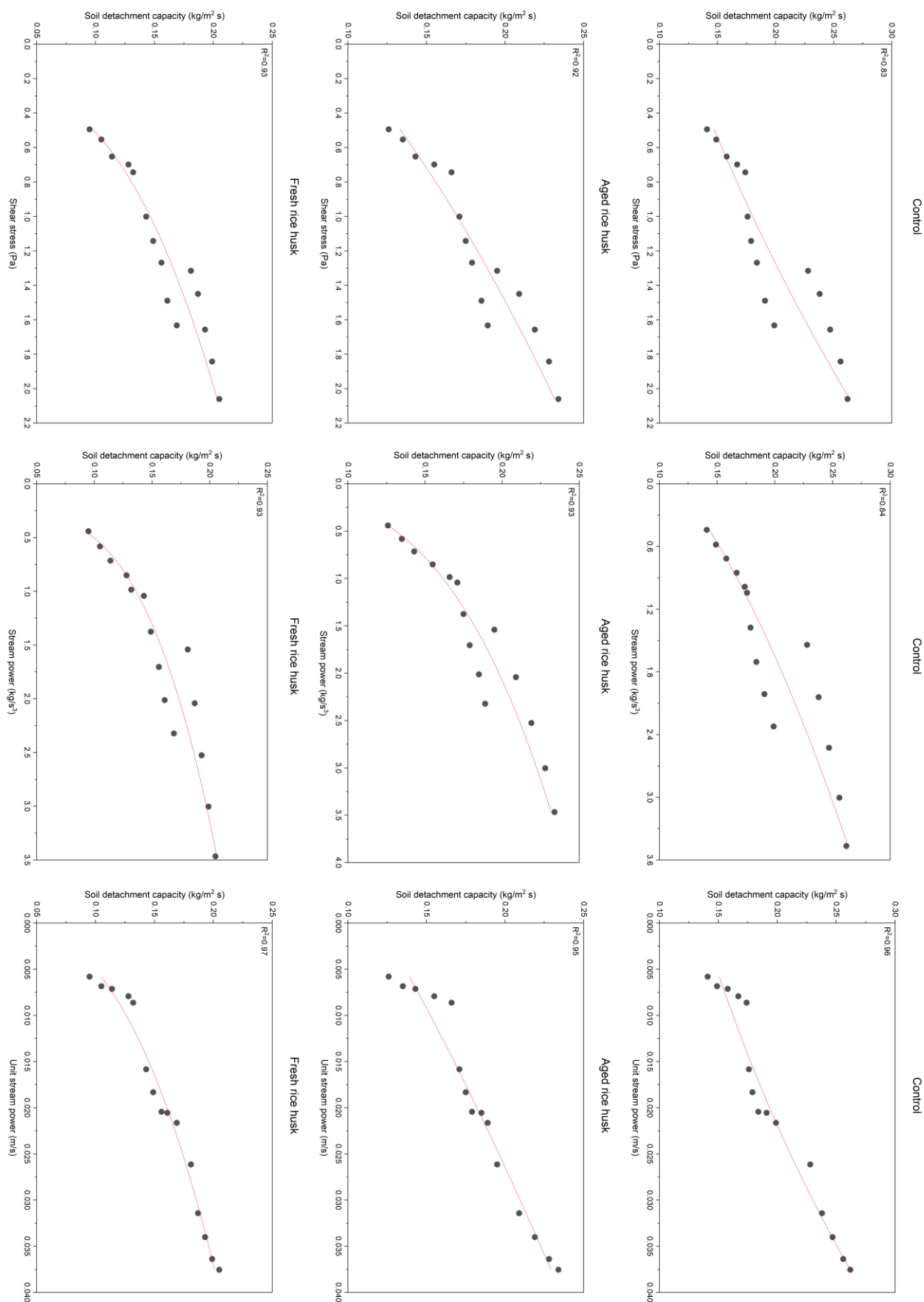


Fig. 6. Non-linear relationships between the soil detachment capacity (D_c) and hydraulic predictors: shear stress (τ), stream power (ω) or unit stream power (ω_u) for control soil and soils treated with nanosilica derived from fresh and aged rice husk in paddy fields (Rasht, Guilan Province, Northern Iran). Lines represent fitted shifted power functions, and corresponding model parameters are reported in Table 4.

Table 4. Coefficients of non-linear regression models describing the relationship between the soil detachment capacity (D_c) and hydraulic predictors: shear stress (τ), stream power (ω) or unit stream power (ω_h) for control soil and soils treated with nanosilica derived from fresh and aged rice husk in paddy fields (Rasht, Guilan Province, Northern Iran). Model parameters (a, b, c) and their standard errors are reported for the shifted power function ($D_c = a(x - b)^c$), along with the coefficient of determination (R^2). Parameter b represents the critical hydraulic threshold; however, negative or highly uncertain values indicate limitations in threshold identification and should be interpreted with caution. Negative values of parameter b are not physically meaningful and reflect statistical uncertainty in threshold estimation.

	Soil condition	Coefficients						R^2
		$a-a'-a''$		$b-b'-b''$		$c-c'-c''$		
		Value	Standard error	Value	Standard error	Value	Standard error	
Dependent variable (D_c , kg/m ² s)	<i>Independent variable (τ, Pa)</i>							
	Control	0.001	0.03	−6.069	31.074	2.726	11.592	0.80
	Aged rice husk	0.137	0.081	−0.462	1.076	0.571	0.401	0.90
	Fresh rice husk	0.162	0.016	0.228	0.224	0.38	0.125	0.92
	<i>Independent variable (ω, kg/s³)</i>							
	Control	0.127	0.09	−0.835	1.736	0.501	0.374	0.82
	Aged rice husk	0.164	0.016	0.048	0.291	0.282	0.074	0.92
	Fresh rice husk	0.148	0.009	0.25	0.152	0.285	0.058	0.92
	<i>Independent variable (ω_h, m/s)</i>							
	Control	0.523	101.233	−0.921	11.808	16.407	205.356	0.95
	Aged rice husk	1.511	1.393	−0.023	0.026	0.672	0.428	0.94
	Fresh rice husk	0.658	0.181	−0.001	0.004	0.362	0.09	0.96

of aggregation state and structural organization rather than direct nanoparticle size measurements. The high increase after ageing suggests that prolonged storage modifies the physico-chemical characteristics of the silica-organic matrix within the husk structure. This statement agrees with Kalapathy et al. (2000) and Yalcin and Sevinc (2001), who showed that thermal and chemical treatments applied to rice husk ash produce silica with variable crystallinity and aggregation states depending on previous conditions. From a mechanistic perspective, nanosilica may enhance aggregation through mechanisms such as siloxane bonding, increased surface interactions, and pore-scale structural rearrangement. Ageing presumably enhances the gradual oxidation of organic constituents and structural rearrangement of amorphous silica compounds. Exposure of biomass to moisture and its degradation due to the microbial population during storage may facilitate condensation reactions among silanol (Si-OH) groups; this enhances particle coalescence and leads to the formation of larger aggregates before calcination (Brinker and Scherer, 1990; Iler, 1979). When the same acid-leaching and thermal protocols are subsequently applied (as in this study), these pre-aggregated structures are less susceptible to fragmentation, resulting in coarser nanosilica particles (Wang et al., 2012). In contrast, fresh husk retains a more heterogeneous organic-silica interface, which, after acid digestion and combustion, produces finer and more dispersed silica particles with a higher specific surface area.

The noticeably higher standard deviation observed for nanosilica from aged rice husk compared to the production from fresh biomass further indicates structural heterogeneity induced by ageing. This heterogeneity may reflect uneven degrees of silica polymerization and partial crystallization. Wang et al. (2012) demonstrated that substrate composition strongly influences nanoparticle morphology even under controlled synthesis conditions. This reinforces the interpretation that the resulting apparent particle size distribution is predominantly driven by intrinsic biomass characteristics rather than extraction protocol (Kalapathy et al., 2000).

These findings extend previous work on rice husk-derived silica by explicitly associating biomass ageing with nanoscale particle variability in biomass structure. While earlier studies primarily focused on optimizing extraction methods (Kalapathy et al., 2000; Wang et al., 2012), the results of this study indicate that raw material condition is also critical in designing soil amendments based on nanosilica. This insight is particularly important for agricultural systems, where residues are frequently stored for long periods before processing.

4.2 Effects of soil treatment with nanosilica from fresh and aged rice husk on aggregate stability and bulk density

Soil treated with fresh husk nanosilica exhibited increases in WSA and BD by 106% and 7.4%, respectively, compared to control soil. The increases compared to the aged biomass were 86% and 6.3%. These differences are not merely statistical but reflect noticeable alterations in soil structural architecture.

Aggregate stability is governed by physico-chemical bonding forces among clay particles, organic matter, polyvalent cations, and mineral surfaces (Bronick and Lal, 2005; Six et al., 2004). The finer nanosilica particles derived from fresh husk should have enhanced aggregation through complementary mechanisms: firstly, by surface area-driven bonding, considering that smaller particles have greater specific surface area, which increases contact points with clay particles and organic colloids, and enhances van der Waals' interactions and hydrogen bonds (Kah et al., 2018; Six et al., 2004); and, secondly, by siloxane bridge formation due to condensation of silanol groups on nanosilica surfaces with hydroxyl groups on clay minerals, forming siloxane (Si-O-Si) linkages that reinforce inter-particle cohesion (Brinker and Scherer, 1990; Iler, 1979); and, third, by micro-pore filling and structural rearrangement, based on occupation of inter-aggregate voids by finer nanosilica, which promotes hierarchical aggregation and reduces macro-porosity collapse under wetting (Bronick and Lal, 2005; Six et al., 2000b). It is worth noting that the improvement in WSA may be beneficial

since this enhances the soil's resistance to slaking during rapid wetting, a critical factor in paddy soils subjected to irrigation and rainfall cycles (Arunrat et al., 2022; Chen et al., 2013).

The improved pore connectivity and a more stable aggregate framework may explain the reduction in bulk density under treatment with nanosilica from fresh biomass. Similar reductions in bulk density following silica-based amendments have been attributed to enhanced flocculation and microaggregate stabilization (Six et al., 2000a). In contrast, the aged husk nanosilica, characterized by a larger apparent particle size, presumably acted more as a coarse filler rather than a true nano-additive bridging soil particles, and this may explain its intermediate performance.

The changes in physical properties after nanosilica amendments align with the broader soil structural theory, which states that micro-scale bonding processes directly control macro-scale mechanical resistance (Bronick and Lal, 2005).

4.3 Effects of soil treatment with nanosilica from fresh and aged rice husk on soil detachment capacity

The flume experiments revealed that the aged husk can reduce soil detachment capacity by less than 10% compared to the untreated soil. In contrast, the addition of fresh husk leads to a reduction of D_c by approximately 21% in particle detachment compared to the control and 14.5% compared to the aged biomass. These reductions are within the range reported in previous studies on soil amendment, where organic or mineral conditioners typically reduce detachment rates by 10-30% under comparable hydraulic conditions (e.g., Knapen et al., 2007; Morgan, 2009a). In absolute terms, the D_c values measured in the present study are consistent with those commonly reported for agricultural soils under rill-flow conditions (Nearing et al., 1997), confirming that the experimental setup reproduced realistic erosive responses.

From a mechanistic standpoint, nanosilica modifies cohesive strength between primary particles, alters micro-topography and surface roughness, and influences pore-scale flow redistribution, thus affecting local concentration of shear stress (Knapen et al., 2007; Morgan, 2009; Nearing et al., 1997). Thus, nanosilica may not suppress hydraulic control but changes the soil mechanical response to applied hydraulic energy. The progressive reduction in D_c from control to aged and fresh husk treatments confirms enhanced aggregate stability and increased resistance to particle entrainment, supporting the concept that soil structural properties are key predictors of erosion resistance (Six et al., 2000b).

4.4 Estimation of erodibility parameters and critical shear stress, stream power and unit stream power in soils treated with nanosilica from fresh and aged rice husk

The relationship between D_c and the hydraulic predictors was described using a shifted power function, which provided satisfactory fits. Across the treatment gradient, the soil erodibility (parameters a , a' and a'') and critical values of the hydraulic predictors generally increased (b , b' and b''). The latter increase suggests a progressive rise in the effective hydraulic threshold required to initiate detachment - consistent with improved soil structural resistance - presumably due to improved aggregation and increased surface protection induced by the rice husk treatments.

At the same time, the higher values of parameters a , a' and a'' indicate a steeper increase in D_c once the threshold is exceeded. This implies that, although treated soils require greater hydraulic

forcing to initiate erosion, detachment rates can rise rapidly after structural bonds are disrupted. This increase presumably reflects the breakdown of stabilized aggregates or localized concentration of hydraulic forces (Hu et al., 2015). However, given the potential interdependence between parameters in shifted power-law models, this trend should be interpreted with caution.

In several cases, especially under control conditions, the fitted parameters b , b' and b'' , theoretically representing the critical hydraulic threshold, assumed negative values or were associated with very large standard errors. This suggests that the three-parameter shifted power function may be over-parameterized for the available dataset. Since a critical shear stress or stream power cannot be negative from a physical point of view, these results indicate that the regression procedure did not identify a statistically significant threshold within the range of applied hydraulic forces; moreover, a strong interdependence among all coefficients under a given soil condition is evident. The large uncertainty associated with these parameters suggests that detachment initiated at very low hydraulic stresses and that the model effectively behaved as a simple power function without a clear threshold. In such cases, fixing to zero or independently estimating the critical parameter from incipient motion criteria could provide more stable parameter estimates and a clearer physical interpretation. Overall, the observed treatment effects should be interpreted primarily with caution, that is in terms of shifts in the overall detachment response rather than as precise changes in a sharply defined critical threshold.

4.5 Study limitations, practical implications, and future research directions

Some limitations should be acknowledged in this study. Firstly, experiments were conducted under controlled flume conditions, which allow proper control of hydraulic variables (Ma et al., 2022; Wang et al., 2019), but cannot fully reproduce rainfall impact or field-scale heterogeneity. Secondly, only one soil type (paddy soil from northern Iran) was tested, limiting extrapolation to soils with different textures or mineralogies. Thirdly, nanosilica was applied at a single rate (2% w/w), while the response to other doses was not explored. Fourthly and finally, long-term environmental implications, including potential interactions with soil microbiota and nutrient cycling, were not assessed.

Despite these limitations, the findings provide important practical implications. More specifically, fresh rice husk produces nanosilica with higher performance on soil stabilization, which indicates that biomass condition is important: farmers and processing facilities should prioritize recently harvested residues when producing silica amendments. Secondly, using rice husk waste for nanosilica is in line with sustainable residue management and reduces the need for synthetic stabilizers, which targets circular economy integration. Thirdly, in intensively tilled and seasonally bare paddy fields, nanosilica application could reduce erosion risk during irrigation and rainfall events, contributing to a reduction in sediment and nutrient loss. Furthermore, about soil structural rehabilitation, nanosilica may serve as a complementary strategy integrating organic amendments for restoring degraded soil structure.

However, there is a need for further studies that, in assessing the effectiveness of rice husk-derived amendments with different ages, should: i) evaluate field-scale performance under natural rainfall conditions, and test multiple soil textures and mineralogies; iii) investigate long-term stability of nanosilica-induced aggregation, preferably exploring optimization of

apparent particle size distribution and application rates. Cost-benefit analyses would also be welcome to determine economic feasibility for farmers. Finally, integrating nanosilica amendments into erosion prediction models could improve parameterization of soil erodibility coefficients under amended conditions.

5 CONCLUSION

This study indicates that the rice husk ageing is a critical condition of nanosilica characteristics and its effectiveness as a soil-stabilizing amendment. The essential indications of the study are, at least under the specific experimental conditions: i) storage induces significant physico-chemical changes altering silica morphology before extraction; ii) finer nanosilica derived from fresh husk significantly increases WSA and reduces BD compared to both aged husk treatment and untreated soils; iii) application of nanosilica reduces soil detachment capacity by up to 20% relative to the control, and fresh husk-derived nanosilica is more effective for erosion mitigation; and iv) the nanosilica treatments shifted the hydraulic response toward higher effective initiation thresholds, indicating enhanced structural resistance to erosion while preserving a non-linear detachment behavior once hydraulic forcing exceeds soil strength.

Overall, the study provides mechanistic evidence that nanoscale particle characteristics influence aggregate formation, BD, and particle detachment, thus filling the gap between material science and soil erosion theory. Incorporating fresh agricultural residues into nanosilica production increases the performance of soil amendment towards effective erosion control strategies. By demonstrating that biomass ageing affects both nanoparticle characteristics and hydraulic erosion parameters, this research advances our understanding of how bio-derived nanomaterials can be optimized for sustainable soil conservation. The findings of this study should be interpreted within the specific experimental conditions considered, namely a single paddy soil type and a fixed nanosilica application rate. Therefore, extrapolation of the results should be limited to similar soil types and environmental conditions. Further research is required to evaluate the applicability of these findings across different soil textures, climatic conditions, and management practices.

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Consent for publication. Not applicable.

Availability of data and material. The datasets generated during and/or analyzed during the current study are not publicly available due to the need to protect authors' intellectual work but are available from the corresponding author on reasonable request.

Competing interests. The authors have no competing interests to declare that are relevant to the content of this paper.

Author contributions. All authors contributed to the study

conception and design. Material preparation, data collection and analysis were performed by Misagh Parhizkar. The first draft of the manuscript was written by Misagh Parhizkar, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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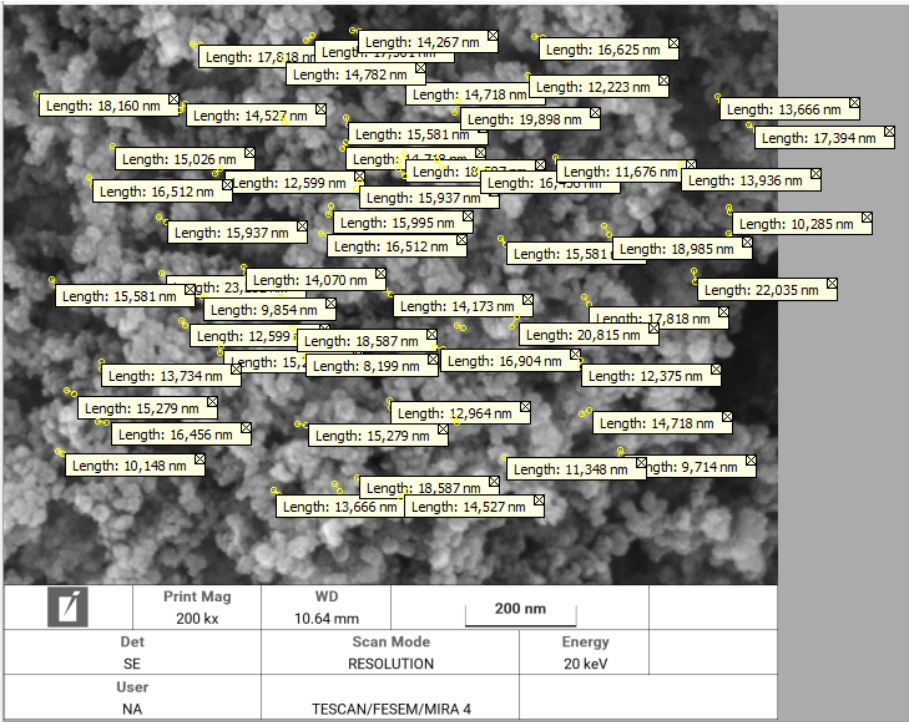
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SUPPLEMENTARY MATERIAL

Fresh rice husks



Aged rice husks

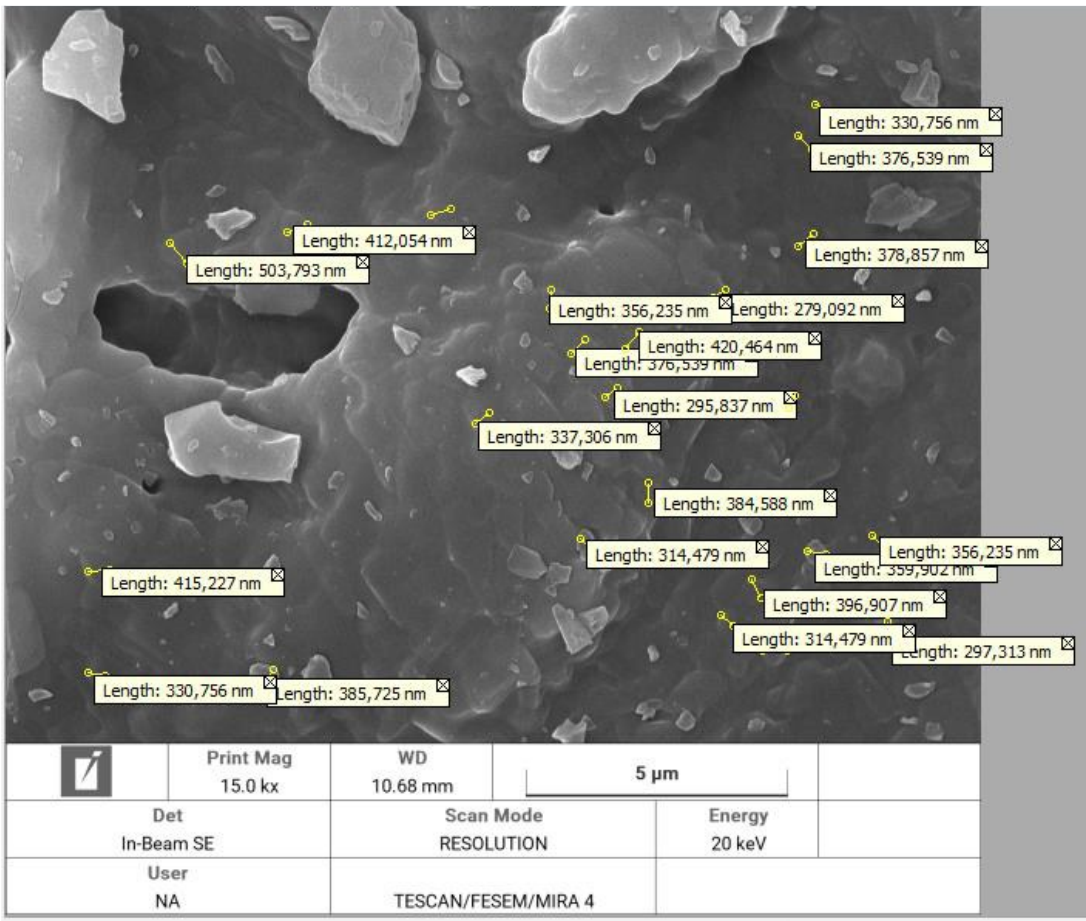


Fig. 1SM. Fourier-Transform Infrared Spectroscopy (FESEM) analysis of nanosilica extracted from fresh and aged rice husks and tested in this study.

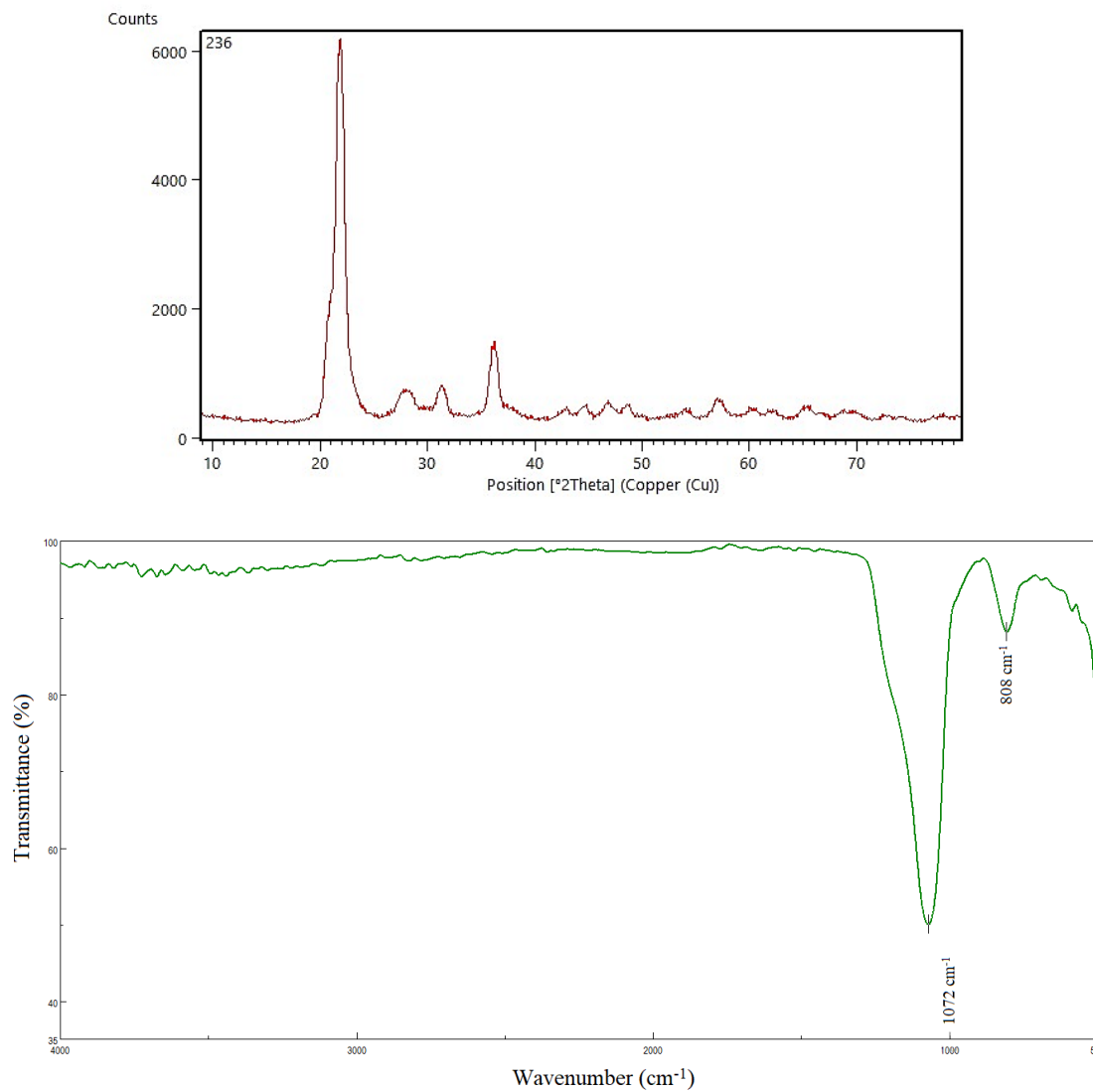


Fig. 2SM. XRD and FTIR analyses of nanosilica extracted from fresh rice husk.

Table 1SM. Purity of silica nanoparticles (by XRF) derived from fresh rice husk.

Chemical compounds	Percentage
Al ₂ O ₃	0.44
Fe ₂ O ₃	0.14
SiO ₂	98.45
CaO	0.13
K ₂ O	0.84
L.O.I.	0.00

Note: L.O.I. = loss on ignition.