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Nanosilica synthesis from rice husk and application for soaking seeds

H X Nguyen*, N T T Dao, H T T Nguyen and A Q T Le

Faculty of Environmental Sciences, VNU University of Science, Vietnam National University, Hanoi, Vietnam

E-mail: huannx@hus.edu.vn

Abstract. Nanosilica was synthesized from rice husk. At first, the rice husk ash was obtained by annealing at temperatures 700°C for 6 hours, then nanosilica particles were extracted by using 0.5N NaOH solution followed by forming gel with HCl acid solution. The structural features of the products were characterized by techniques including Energy dispersive X-ray, Field Emission Scanning Electron Microscopes, Fourier Transformation Infrared, Fourier Transformation Infrared. The results showed that the SiO₂ nanoparicles have average size about 30-100 nm and >98 % purity. The present experiment was conducted to test the beneficial effects of nanosilicon on the seed germination. Seeds (BC rice, Bachthom rice and bean) were analyzed with regard to germination percentage (GP), mean germination time, seed germination index, seed vigour index to investigate the efficiency of nanosilica. Among the treatments, the addition of 40 mg/L nanosilica for BC rice seed, 60 mg/L for Bachthom rice seed showed the most promising results that yielded significant differences from the control in terms. Compare to GP of BC and Bachthom sample after treatment (100%), GP of sample with no-nanosilica are 93.33% and 53.33%, respectively. Besides, the samples which added nanosilica have higher average length of shoot and root than blank sample.

1. Introduction

Rice husk ash is the by-product of rice husk, when it burnt in ambient atmosphere with twenty million tons per year by world (Koteswara and Pranav 2011; Soltani et al. 2015). Due to low density and less commercial interest of rice husk, handling as well as transportation it is problematic, which creates disposal and serious environmental problems (Pode 2016). According to the field survey, the average proportion of rice straw burned in the field was around 44%. The total amount of pollutants were emitted as CO₂ (419,889.1 tons), CO (8865.1 tons); NMVOC (3565.6 tons); PM_{2.5} (3466.7 tons); NO_x (1402.1 tons); OC (779.7 tons); CH₄ (263.6 tons); EC (208.7 tons); NH₃ (194 tons); and SO₂ (58.6 tons). According to the experiments, the main element in rice husk ash is silicon (87.7% as SiO₂), followed by potassium (5.4% as K₂O) and phosphorous (3.7% as P₂O₅) so rice husk ash (RHAs) are likely an abundant silica source show a lot promises for science and application for many fields.

In agriculture, silica is considered as an essential micronutrient and plays an important role in plant growth and productivity. In addition, it makes leaf, shoot and root be stronger and improves tolerance to weather conditions and reduces the negative effects of some toxic elements in the soil. In germination period, silica can sterilize and destroy fungi, increase germination rate. And finally, with nanoscale and high surface area, nanosilica (0-100 nm) can pass through the cell membrane easily. Therefore, using nanomaterials, for example, nanosilica is considered as new researching direction to



soaking seeds. Nanosilica can be extracted from chemical or natural materials. nanosilica was produced by various techniques including Stobër technique (Gholami, Salavati-Niasari et al. 2013), sol-gel methods (Le, Thuc et al. 2013), and water-in-oil nanoemulsion system (Park, Oh et al. 2003). However, few studies have been carried out using slow gelation technique and freeze-drying methods (Lu and Hsieh 2012).

In this study, nanosilica is synthesized by sol-gel method from rice husk. This materials were characterized using Energy-dispersive X-ray spectroscopy (EDX), Fourier Transformation Infrared (FTIR) and X-ray dif-fraction (XRD), Scanning electron micrograph (SEM), Field emission scanning electron microscopy (FESEM). In other aim of this study is to investigate the effects of nanosilica particles were assessed to parameters such as germination percentage, germination index, vigor index, mean germination time and average length of shoot and root.

2. Materials and methods

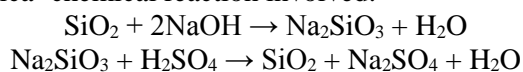
2.1. Materials

The chemicals consisted of hydrochloric acid (HCl, 37%, China), sodium hydroxide (NaOH, ≥96%, China), and distilled water. And rice husk in the Red river delta was used as silica sources.

Rice seeds (Bacthom and BC) and Bean seed for soaking.

2.2. Methods

2.2.1. Nanosilica synthesis method from rice husk. Nanosilica is synthesized by sol-gel method from rice husk. Precipitation of silica- chemical reaction involved:



Nanosilica is synthesized by sol-gel method with 5 main steps:

Washing and dry: Rice husk was washed thoroughly with water to remove the soluble particles, dust, and other contaminants present, whereby the heavy impurities such as sand are also removed. It was then dried in an air oven at about 110°C for 24 hours.

Acid treatment: An acid washing step was used to remove the small quantities of minerals prior to silica extraction from rice husk ash (RHA). Ten grams of RHA sample were dispersed in 60 ml of HCl 1N.

Thermal treatment: A weighed RH as well as RH were subjected to heat treatment to obtain the ash. Samples were burned inside a programmable furnace (Nabertherm controller B 170, Nabertherm GmbH, Lilienthal, Germany) at different temperatures (500, 700, and 1000°C) and optimal temperature 700°C in 6 hours.

Silica extraction: A sample of 2.5 g RHA was stirred in a 250 mL, 0.5N sodium hydroxide solution. The solution was heated in a covered beaker by stirring constantly and was allowed to stand at room temperature then filtered.

Nanosilica preparation: HCl was added until neutralized. The precipitate silica was washed repeatedly with warm, deionized water and then was centrifugated with speed 5000 rpm within 10mins repeated in 3 times. The product is dried at 110°C for 24 hours in the oven and crystallization in a programmable furnace at 450°C in 1 hour. Then crushed them to collect nanosilica.

2.2.2. Soaking seeds method by nanosilica. Three kinds of seed (BC rice, Bacthom rice and bean) were immersed in distilled water, for about 1 hour then they were soaked in different concentration nanosilica. One piece of filter paper was put into each 100 mm x 15 mm Petri dish, and 5 mL of a test solution was added. Seeds were transferred onto the filter paper, with 15 seeds per dish and 1cm or larger distance between each seed (Kikui, 2005). Petri dishes were covered and sealed with tape, placed in an incubator at room temperature (30.2°C). After 3 and 6 days in the dark under room temperature, seed parameters was calculated and measured.

2.2.3. Research on impact of nanosilica on Germination percentage (GP).

$$GP = n/N \times 100\%$$

Where:

n is the total number of seeds germinated.

N represents the total number of tested seeds (Shi, Zhang, Yao, Wu, Sun, & Gong, 2014; Yu, Duan, Xu, Zhang, & Jin, 2014).

2.2.4. Research on impact of nanosilica on Mean germination time (MGT). Mean germination time is an accurate measure of the time taken for a lot to germinate calculated by the expression (A.Ranal et al., 2009).

$$\bar{t} = \frac{\sum_{i=1}^k n_i t_i}{\sum_{i=1}^k n_i}$$

Where:

t_i is time from the start of the experiment to the ith observation (day for the example).

n_i is number of seeds germinated in the ith time (not the accumulated number, but the number correspondent to the ith observation).

k is last time of germination

2.2.5. Research on impact of nanosilica on Germination index (GI). Germination Index is the analysis method that best describes the germination percentage/ speed relationship. The GI appears to be the most comprehensive measurement parameter combining both germination percentage and speed (spread, duration and 'high/low' events).

$$GI = \sum (G_t / D_t)$$

Where:

G_t is the number of seed germinated at t day.

D_t represents the corresponding day of germination (Shi et al., 2014; Muharrem, Gamze, Mehmet, Mehmet, Sevil, Khalid, & Cemalet-Tin, 2008).

2.2.6. Research on impact of nanosilica on Virgo index (VI).

$$VI = \text{mean shoot length (cm)} \times GP \%$$

(AbdulBaki & Anderson 1973; Amooaghaie et al., 2015; Dhindwal, Lather, & Singh, 1991; Siddiqui & AlWhaibi, 2014)

2.2.7. Research on impact of nanosilica on dispersion. The ratio of the intensity of the transmitted light (I) to the intensity if the incident light (I_o) is called transmittance (T).

$$T = I / I_o$$

$$\%T = T * 100$$

2.2.8. Research on impact of nanosilica on keeping moisture. With the different nanosilica concentration (0, 0.1, 0.5, 1.5, 2.5 g), after 3 and 6 days in incubator at 37°C, 5 sample was taken, observed and carried out measuring.

3. Results and discussion

3.1. Characteristic of nanosilica extract from rice husk

3.1.1. Nanosilica extract composition. EDX elemental spectra of rice husk showed major element silicon (Si) and impurities Rhodium (Rh), lead (Pb), iron (Fe) and Arsenic (As). Silica content of the xerogels was estimated from the EDX data based on the assumption that all of the silicon was in the form of silica. The silica content of xerogels produced from washed RH were 99.5%. It is interesting

to note that impurity elements were also present in a lower concentration even detected in the nanosilica. The color of nanosilica was white after synthesis.

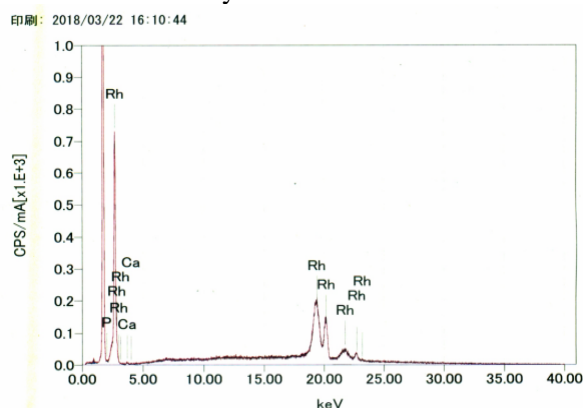


Figure 1. Energy-dispersive X-ray spectroscopy of nanosilica

3.1.2. Nanosilica extract functional group. FTIR spectroscopy was used to detect the presence of functional groups in the silica nanoparticle (SNPs). The major chemical groups present in silica are identified by the FTIR spectra shown in Figure 2. Six functional groups are found in samples: hydroxyl groups, C-H stretching, C=C stretching, Si-O-Si stretching, Si-H groups, and $-\text{OCH}_3$ stretching.

The adsorption peak around 3447.27 cm^{-1} indicates the existence of free hydroxyl groups. In rice husk, the C-H stretching vibration around 2924.02 cm^{-1} indicates the presence of alkane functional group. The C=C stretching vibrations between $1636.44\text{--}1653.91\text{ cm}^{-1}$ indicates alkenes and aromatic functional groups. The peaks around $1077.8\text{--}1097.58\text{ cm}^{-1}$, 800.12 cm^{-1} , and $466.76\text{--}470.37\text{ cm}^{-1}$ correspond to Si-O-Si stretching, Si-H groups, $-\text{OCH}_3$ stretching, respectively. The presence of polar groups on the surface is likely to provide the considerable cation exchange capacity to the adsorbent.

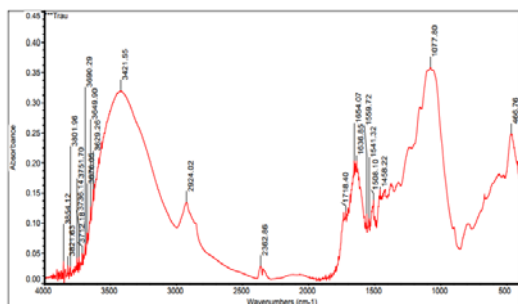


Figure 2. FTIR of rice husk

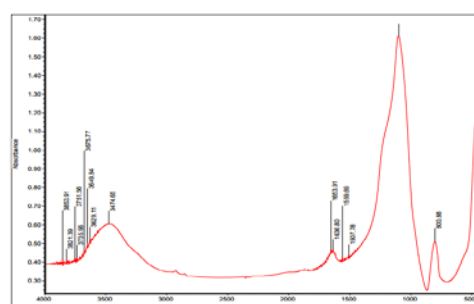


Figure 3. FTIR of rice husk ash

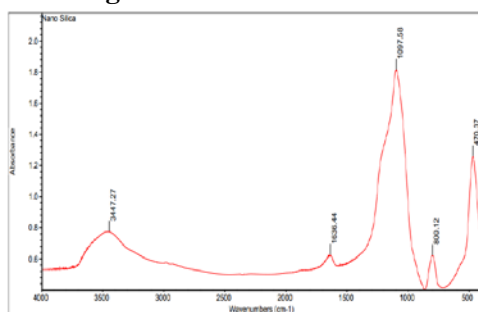


Figure 4. FTIR of nanosilica from rice husk

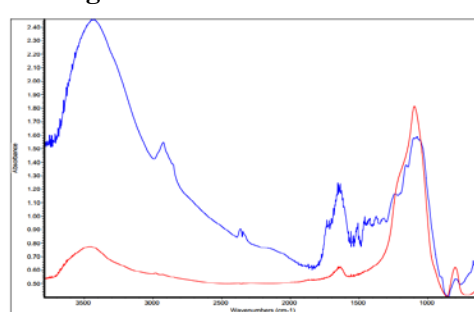


Figure 5. FTIR of comparison rice husk and nanosilica

Compared to rice husk, synthesized nanosilica resulted in a loss of C-H stretching band and a addition of Si-H. These were decreased in the primary functional groups of OH and C=C and increased in silica functional groups of Si-O-Si.

3.1.3. Nanosilica extract morphology and particle size. Figure 6 illustrates the SEM images of SNPs samples. It presented that synthesized nanosilica have spongy structure. Particle size and morphology of synthesized silica were examined by FESEM with high enlargement. It can be observed that SNPs have a circular shape with average particle size of 30-100 nm.

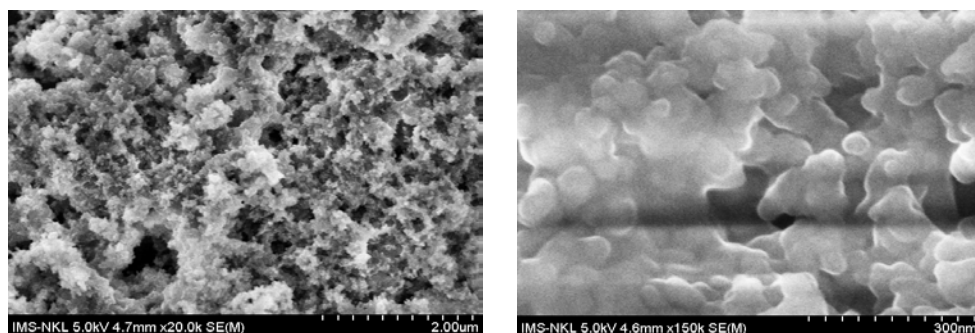


Figure 6. Field emission scanning electron micrograph (FESEM) of nanosilica from rice husk

3.1.4. Nanosilica extract structure. X-ray diffraction pattern of extracted silica is presented in Figure 7. Hill like peak in the range of $2\theta = 21$ to 24 , indicates the absence of any ordered crystalline structure and highly disordered structure of silica and extracted nanosilica is purity.

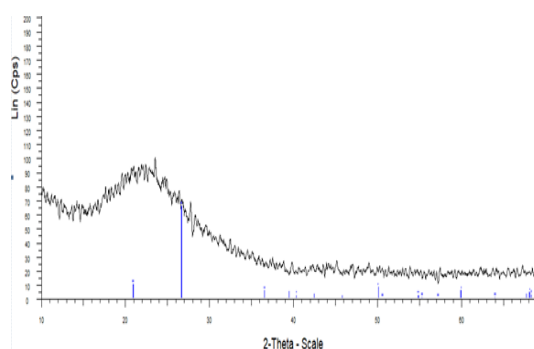


Figure 7. X-ray diffraction pattern of nanosilica

3.1.5. Nanosilica extract dispersion.

Table 1. Results of the transmittance of nanosilica solutions

No.	Dispersing time (hours)	T (%)
1	0	95.134
2	0.5	87.018
3	1	85.631
4	1.5	82.152
5	2	80.582
6	2.5	79.892
7	3	81.440

The result shows that the initially sample which nanosilica is just stirred has the largest transmittance (95.134%). The transmittance decreases while the time increases but at 3 hours, the transmittance raises following time (81.440%). After 2.5 hours, the transmittance is the lowest with 79.892%. It means that at this time, dispersible nanosilica is the best and ability of passing through solution of light is the lowest.

3.1.6. Nanosilica ability of keeping moisture.

Table 2. Remaining water percentage after 3 days and 6 days

No.	Nanosilica mass (gram)	Remaining water percentage (%)	
		3 days	6 days
1	0	44.9	0.99
2	0.1	45.1	15.13
3	0.5	63.98	31.45
4	1.5	77.18	36.65
5	2.5	88.93	47.83

The result shows that about the sample with no nanosilica, water is almost evaporation approximately 100% after 6 days. In while, addition 2.5 g nanosilica for sample keep 89% moisture after 3 days, are more 2 times than blank sample and after 6 days, remaining water percentage is equal 47.83%.

This blank sample is completely different from the rest. When nanosilica concentration is increase, the remaining water percentage after 3 hay 6 days is raise. It proves that nanosilica can keep moisture.

3.2. Impacts of nanosilica on germination parameters

3.2.1. Rice seeds.

Table 3. Effect of nanosilica on rice seed germination

Rice seed type	No.	Conc. (mg/L)	3 days				6 days			
			Root length (mm)	Shoot length (mm)	Root length (mm)	Shoot length (mm)	GP (%)	MGT	GI	VI
BC	1	0	69 ± 1.51	22 ± 0.87	107 ± 1.95	44 ± 0.6	93.33	4.32	13.98	410
	2	10	81 ± 1.66	32 ± 0.5	128 ± 1.27	48 ± 1.4	100	4.27	16.47	480
	3	20	93 ± 0.32	33 ± 0.32	147 ± 1.65	59 ± 1.4	100	4.25	17.00	590
	4	40	94 ± 0.57	38 ± 0.7	196 ± 2.86	72 ± 2.0	100	4.18	18.58	720
	5	60	98 ± 0.95	34 ± 0.85	139 ± 2.10	62 ± 0.8	100	4.30	16.17	620
	6	80	94 ± 0.92	31 ± 0.29	135 ± 0.74	57 ± 1.0	100	4.23	17.25	570
	7	100	92 ± 0.91	30 ± 0.46	130 ± 1.53	54 ± 0.9	100	4.26	16.67	540
Bacthom	1	0	63 ± 1.4	15 ± 1.16	168 ± 1.68	33 ± 1.55	53.33	4.65	5.53	176
	2	10	70 ± 1.1	21 ± 1.14	186 ± 1.5	37 ± 1.56	86.67	4.64	9.33	321
	3	20	95 ± 1.35	23 ± 1.70	222 ± 1.48	42 ± 0.96	93.33	4.50	11.90	392
	4	40	96 ± 1.28	40 ± 2.29	226 ± 1.35	48 ± 1.16	93.33	4.49	13.22	448
	5	60	94 ± 1.38	25 ± 1.35	279 ± 1.42	66 ± 1.81	100	4.37	15.25	660
	6	80	81 ± 1.42	21 ± 1.30	236 ± 1.44	58 ± 1.82	93.33	4.42	12.82	541
	7	100	76 ± 1.25	20 ± 0.81	225 ± 1.53	44 ± 1.57	93.33	4.49	12.27	401

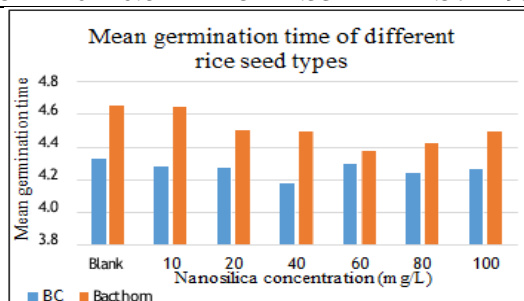


Figure 8. Effect of nanosilica on mean germination time of different rice seed types

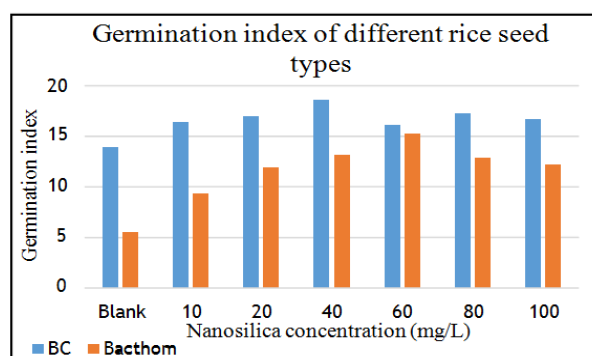


Figure 9. Effect of nanosilica on germination index of different rice seed types

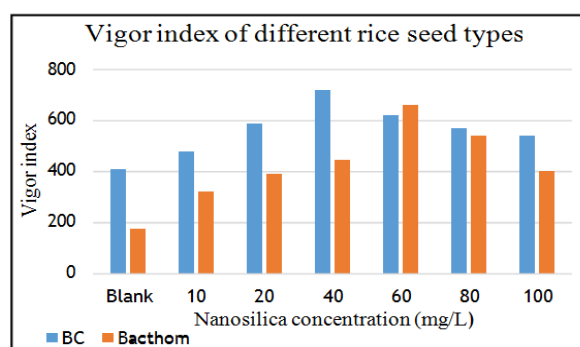


Figure 10. Effect of nanosilica on vigor index of different rice seed types

The shoot and root length change unclearly after 3 days. By 6 days, 40 mg/L is considered as optimal nanosilica concentration for BC length with 72 mm of shoot and 196 mm of root while 60 mg/L is the best for Bacthom with 66 mm of shoot length and 279 mm of root length.

Germination percentage (GP) of BC samples when adding nanosilica all reached to 100%. In comparison with control (53.33%), GP of Bacthom was 100% only at 60 mg/L. The control presents an average germination time (MGT) of 5.41 days for BC and 5.82 days for Bacthom while the treatment with the lowest MGT was observed with the addition of 40 mg/L at an average germination time of 5.19 days of BC and 5.43 days after adding 60 mg/L for Bacthom.

A significant difference on the germination index (GI) was noted starting with the addition of 40 mg/L for BC (18.58) and 60 mg/L for Bacthom (15.25) relative to the control (13.98 for BC, 5.53 for Bacthom).

Vigor index (VI) was significantly increased by 720 for BC and 660 for Bacthom with the addition of optimal concentration compared to control.

It indicated clearly that the 40 mg/L of nanosilica for BC and 60 mg/L for Bacthom were found to be the best for the growth enhancement of seed.

3.2.2. Bean seed.

Table 4. Effect of nanosilica on bean seed germination

No.	Conc. (mg/L)	3 days					
		Root length (mm)	Shoot length (mm)	GP (%)	MGT	GI	VI
1	0	36 ± 1.12	45 ± 1.3	93.33	2.17	19.67	420
2	10	39 ± 1.4	48 ± 1.24	100	2.15	21.5	310
3	20	41 ± 1.35	52 ± 1.62	100	2.13	21	560
4	40	42 ± 1.4	56 ± 1.2	100	2.15	21.5	750
5	60	47 ± 1.55	53 ± 1.41	100	2.16	21	410
6	80	43 ± 1.3	49 ± 1.53	100	2.18	20.5	540
7	100	44 ± 1.6	48 ± 1.36	100	2.19	20	730

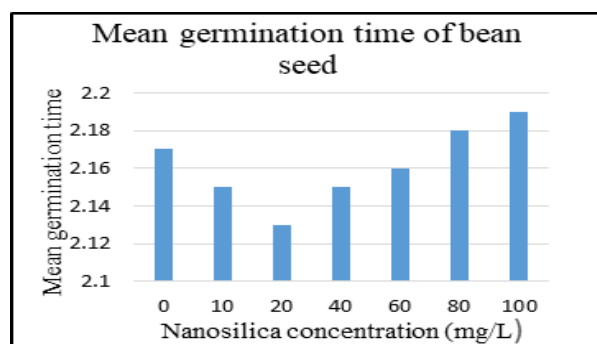


Figure 11. Effect of nanosilica on mean germination time of bean seed

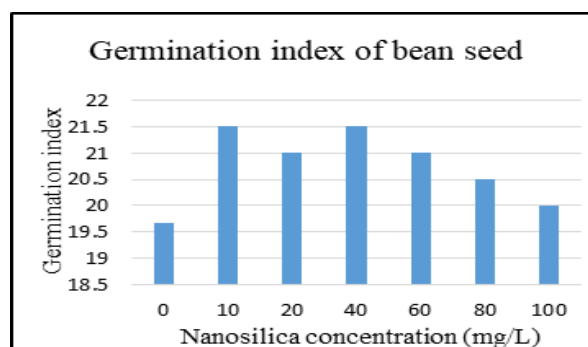


Figure 12. Effect of nanosilica on germination index of bean seed

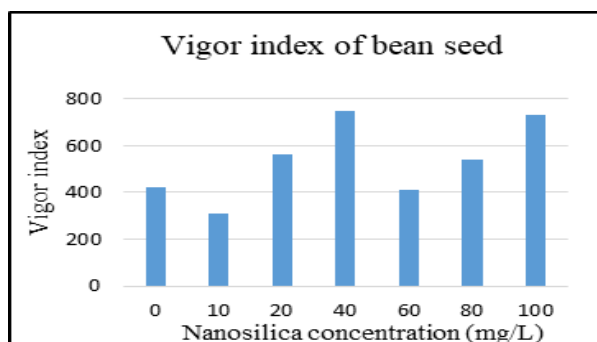


Figure 13. Effect of nanosilica on vigor index of bean seed

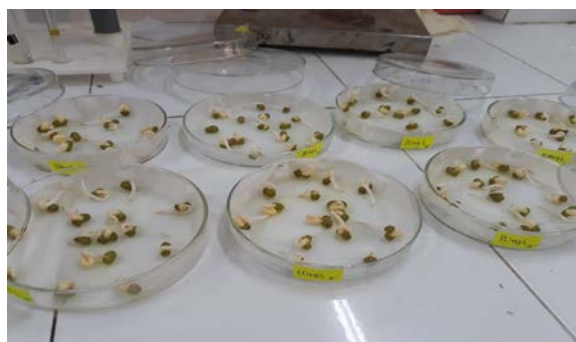


Figure 14. Application of nanosilica for soaking bean seed after 3 days

100% number of bean seeds germinated compared with 93.33% of control. At 40 mg/L, shoot length (56 mm) is maximum but root length (47 mm) is maximum at 60 mg/L. MGT (2.13) is the lowest and at 20 mg/L, MGT (2.19) is the highest and higher than MGT of blank sample. Vigor index of bean seeds was changeable with the addition of nanosilica powder which is highest (750) with 40 mg/L nanosilica. GI increased rapidly from 19.67 and reached to 21.5 after adding 10 and 40 mg/L nanosilica.

4. Conclusion

Synthesized nanosilica are purity ($\approx 98\%$) and have spongy and disordered structure and a circular shape with average particle size of 30-100 nm by modern techniques such as EDX, FTIR, SEM, FESEM, XRD. With nanoscale, silica have the potential ability of passing the cell membrane of plant and can be used as an immediately utilizable source for soaking seeds.

Addition of 40 mg/L nanosilica for BC rice seeds, 60 mg/L for Bacthom rice seeds showed the most promising results that yielded significant differences from the control in terms of average length, GP, MGT, GI, VI.

For bean seeds, at addition of 40 mg/L nanosilica, MGT of (2.13) is lower than MGT of blank sample (2.17); GP (100%), VI (750) are the larger than blank ones. In while, GI at 10 mg/L and 40 mg/L are all equal to 21.5.

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