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# Determination of Calcium Carbonate Equivalent of Rice Husk Ash

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#### Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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# ABSTRACT

Rice husk ash is one of the widely available agricultural wastes in rice producing countries, with potential to replace limestone and also supply some of the nutrients which are beneficial for crop growth. This study was conducted to determine the effective calcium carbonate equivalent (ECCE) of rice husk ash added to an acid soil. The design used in this study was completely randomized block design with 12 treatments and 3 replications. An incubation experiment was carried out in the post graduate laboratory of Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore, during Feb - March 2022. The incubation experiment was conducted for 4 weeks with eight levels of reagent grade CaCO<sub>3</sub> (0.0, 0.5, 1.0, 1.5, 2.0, 3.0, 4.0 and 5.0g kg<sup>-1</sup> soil) and four levels of rice husk ash equivalent to 2.0, 3.0, 4.0 and 5.0 g CaCO<sub>3</sub> kg<sup>-1</sup> soil and analysed for pH at weekly interval. Results showed that soil pH increased in all the treatments received CaCO<sub>3</sub> or rice husk ash with advancement of incubation period. Application of CaCO<sub>3</sub>5.0 g kg<sup>-1</sup> soil recorded significantly highest soil pH at all stages of incubation followed by CaCO<sub>3</sub> 4.0 g kg<sup>-1</sup> soil and RHA 48.5 g kg<sup>-1</sup> soil which were statistically on par. The TCCE (determined by titration method) and ECCE (determined by incubation with the soil) of rice husk ash used in this experiment were 10.3% and 6.41% respectively. Results of the present study showed that rice husk ash can be used as an amendment for acid soil reclamation.

Keywords: Acid soil; rice husk ash; neutralization of soil acidity; calcium carbonate equivalent.

# **1. INTRODUCTION**

In India acid soils cover around 48 to 49 million hectares arable land of which 25 mha have pH below 5.5 and 23 mha have pH between 5.6 and 6.5, especially in Tamil Nādu around 4.85 mha [1]. Although we can't modify the acidic nature of the soil, the least we can do is to make the soil suitable for agriculture. Wherever rainfall exceeds evapotranspiration, bases such as calcium, magnesium and potassium and their salts are displaced by  $H^+$  derived from the dissolution of  $CO_2$  in  $H_2O$ . The toxicity of aluminium and manganese. low pH and deficits of certain important elements like calcium, phosphorus. magnesium. boron and molybdenum are all major growth limiting factors in acid soil [2]. Liming with various liming materials is one way for acid soil management.

Rice husk ash is a common agricultural waste in rice-producing countries, with the ability to replace limestone and provide some nutrients that are favourable to crop growth. Rice husk is used as boiler fuel in rice mills which generate rice husk ash as a waste. In general, every 100 kg of husk burnt in a boiler will yield about 25 kg of RHA [3]. RHA is most easily available because of the wide spread prevalence of rice mills and these rice husk converted into rice husk ash by the process of combustion. India has produced around 31 million tons of rice husk and thus generated 4.65 - 5.58 million tons (15-18% of rice husk) of Rice Husk Ash (RHA) [4]. There are about 3000 modern rice mills in Tamil Nadu which generate considerable husk and rice husk ash every day. Poor management of this waste material leads to environmental problems. Hence utilization of such waste material not only farmers benefits but also protects our environment.

Rice husk ash is alkaline in nature and can raise the pH of an acidic soil, making it appropriate for crop production. Physical and chemical aspects of soil are essential agricultural phenomena that influence the soil's productive ability. Rice husk ash is a promising and environmentally favourable agricultural resource. RHA not only adjust soil pH and also supply nutrients to crops because it contains  $CO_2 - 0.10\%$ ,  $SiO_2 - 89.90\%$ ,  $K_2O - 4.50\%$ ,  $P_2O_5 - 2.45\%$ , CaO - 1.01%, MgO -0.79%, Fe<sub>2</sub>O<sub>3</sub> - 0.47%, Al<sub>2</sub>O<sub>3</sub> - 0.46%, MnO -0.14% [4]. It can also keep the soil's physical qualities, such as bulk density and total porosity in good shape. Total Calcium Carbonate Equivalent (TCCE) as estimated by titration method is the capacity of rice husk ash to neutralize acidity. The Effective Calcium Carbonate Equivalent (ECCE) is the fraction of TCCE that reacts with soil acidity during reaction with soil. The aim of this work was to study the influence of rice husk ash application on soil pH and to determine CCE of rice husk ash added to an acid soil.

# 2. MATERIALS AND METHODS

An incubation experiment was carried out in the post graduate laboratory of Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore during Feb – March 2022 to determine the effective calcium carbonate equivalent of rice husk ash and the potential of using rice husk ash as an amendment for acid soil reclamation. The soil for conducting the incubation experiment was collected from research farm of Horticultural Research Station, Woodhouse farm, Ooty. Soil samples were air dried and ground to pass through 2 mm sieve. Field capacity moisture content of the soil was analysed [5].

Rice husk ash was collected from a modern rice mill and analysed for Total calcium carbonate equivalent (TCCE) by titration method [6]. Total calcium carbonate equivalent (TCCE) or Neutralizing Value of the rice husk ash was 10.3 %. RHA pH was analysed in 1:10 ratio (RHA: deionized water) by using pH meter [7]. RHA was digested in Microwave digestion system (HNO<sub>3</sub>: HCI: HF in 6: 2: 2 ratio) prior to analysis of C, Si, N, P, K, Ca, Mg, S, Na, Al, Cu, Fe, Mn and Zn content in ICP-OES instrument (Inductively Coupled Plasma Optical Emission Spectroscopy) [8].

One hundred gram of soil was incubated for 4 weeks with eight levels of reagent grade CaCO<sub>3</sub> (0.0 (T<sub>1</sub>), 0.5 (T<sub>2</sub>),1.0 (T<sub>3</sub>), 1.5 (T<sub>4</sub>), 2.0 (T<sub>5</sub>), 3.0 (T<sub>6</sub>), 4.0 (T<sub>7</sub>) and 5.0 (T<sub>8</sub>) g kg<sup>-1</sup> soil) and four levels of rice husk ash (T<sub>9</sub>, T<sub>10</sub>, T<sub>11</sub>and T<sub>12</sub>) equivalent to 2.0, 3.0, 4.0 and 5.0 g CaCO<sub>3</sub> kg<sup>-1</sup> soil. The levels of rice husk ash for T<sub>9</sub>, T<sub>10</sub>, T<sub>11</sub> and T<sub>12</sub>were 19.4, 29.1, 38.8 and 48.5g kg<sup>-1</sup> soil (10.3% TCCE). The experimental units consisted of 12 treatments arranged in a completely randomized block design with three replications. The moisture content of the samples was maintained at field capacity by correcting the

moisture loss periodically using distilled water. Destructive soil sampling was done at weekly interval and analysed for pH. Soil pH was determined in 1:2.5 soil: water suspension.

The ECCE of rice husk ash was derived from the relationship between the levels of pure calcium carbonate applied and pH after fourth week of incubation. The best fit regression model is quadratic polynomial model. Four ECCE values (g CaCO<sub>3</sub> kg<sup>-1</sup>) were calculated corresponding to the pH for the different levels of rice husk ash after fourth week of incubation. The ECCE values expressed in percentage using the following equation [9] and ECCE of rice husk ash in the present study was calculated by taking average of the four values.

ECCE (%) = 
$$\frac{\text{ECCE Estimated } (g \text{ CaCO } 3 \text{ kg} - 1)}{\text{Amount rice husk ash } (g \text{ kg} - 1)} X 100$$

The data obtained from the experiment were subjected to statistical analysis using AGRESS software version 7.01. The level of significance used were P< 0.05. Critical difference (CD) values were calculated for the P< 0.05 whenever "F" test was found significant [10].

# 3. RESULTS AND DISCUSSION

The physico chemical properties of the experimental soil are given in Table 1. The soil was having clay loam texture, acidic in reaction and non-saline. Organic carbon content of the soil was high with available N, P and K content of medium, medium and high status respectively. Sulphur and micronutrients status was found to be sufficient.

The chemical composition of rice husk ash used in this experiment are given in Table 2. The pH of the rice husk ash was 8.3. At 300-350°C the destruction of cellulose and hemicellulose in rice husk produce organic acids and phenolic substances. After 350°C, alkali salt separated in from those organic matrixes which in turn increase the pH of rice husk ash [11].

# 3.1 Effect of Rice Husk Ash on Soil pH

The most important property influenced by amendment for soil acidity reclamation is soil pH. Soil pH increased in all the treatments received CaCO3 or rice husk ash with advancement of incubation period (Table 3). Among the treatments, application of  $CaCO_3 5.0 \text{ g kg}^{-1}$  soil (T<sub>8</sub>) recorded significantly highest soil pH at all stages of incubation followed by  $CaCO_3 4.0 \text{ g} \text{ kg}^1$  soil (T<sub>7</sub>) and RHA <sub>48.5</sub> g kg<sup>-1</sup> soil (T<sub>12</sub>) which were statistically on par. The magnitude of increase in soil pH was high with calcium carbonate application when compared to rice husk ash which might be due to the relatively higher neutralizing value of calcium carbonate. At all stages of incubation, lowest soil pH was registered in control  $(T_1)$ . The pH of the rice husk ash was 8.3. Rice husk ash used in this experiment had Total Calcium Carbonate Equivalent (TCCE) of 10.3%. Because of the high pH, rice husk ash acted as a liming material and increased the soil pH. Increase in soil pH with rice husk ash application was already reported [11-15].

Even though the levels of rice husk ash applied was equivalent to 2.0, 3.0, 4.0 and 5.0 g  $CaCO_3$  kg<sup>-1</sup> soil, RHA could not produce the same effect on soil pH as that of equivalent quantity of lime. This might be due to the high organic matter content of RHA which reduce the efficiency of RHA calcium carbonate [12].

Table 1. Properties of experimental soil used in incubation experiment

рΗ	EC	Organic	Texture	Available			Available	DTPA extractable			
		С		Ν	Р	Κ	S	Fe	Zn	Mn	Cu
	dSm⁻¹	g kg⁻¹		kg ha	1		mg kg⁻¹				
4.92	0.26	30.0	Clay loam	442	45.7	568	18.2	43.2	1.30	10.3	1.92

Table 2. Chemical composition of the rice husk ash used in this exper	iment
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рΗ	NV*	Ν	Ρ	Κ	Si	Са	Mg	S	Na	Al	Fe	Zn	Mn	Cu
	% mg kg <sup>-1</sup>													
8.3	10.3	0.09	0.26	0.72	40.5	0.39	0.27	0.15	1.22	0.88	1591	49.4	426	13.2
* NV- Neutralizing value														

Treatments	l week	ll week	III week	IV Week	
T <sub>1 -</sub> Control	4.95	5.05	5.02	5.09	
$T_2$ - CaCO <sub>3</sub> 0.5 g kg <sup>-1</sup> soil	5.10	5.14	5.17	5.21	
$T_3$ - CaCO <sub>3</sub> 1.0 g kg <sup>-1</sup> soil	5.37	5.41	5.46	5.52	
$T_4$ - CaCO <sub>3</sub> 1.5 g kg <sup>-1</sup> soil	5.47	5.52	5.58	5.64	
$T_5 - CaCO_3 2.0 \text{ g kg}^{-1}$ soil	5.54	5.61	5.68	5.74	
$T_6$ - CaCO <sub>3</sub> 3.0 g kg <sup>-1</sup> soil	5.75	5.81	5.88	5.93	
$T_7 - CaCO_3 4.0 \text{ g kg}^{-1}$ soil	5.86	6.03	6.11	6.17	
$T_8$ - CaCO <sub>3</sub> 5.0 g kg <sup>-1</sup> soil	5.95	6.12	6.18	6.23	
T <sub>9</sub> - RHA 19.4 g kg⁻¹ soil	5.36	5.42	5.50	5.56	
T <sub>10</sub> - RHA 29.1 g kg <sup>-1</sup> soil	5.52	5.60	5.67	5.71	
T <sub>11 -</sub> RHA 38.8 g kg <sup>-1</sup> soil	5.63	5.68	5.75	5.87	
T <sub>12 -</sub> RHA 48.5 g kg <sup>-1</sup> soil	5.76	5.79	5.89	5.99	
SEd	0.12	0.12	0.12	0.12	
CD (P=0.05)	0.25	0.25	0.25	0.26	

Table 3. Effect of CaCO<sub>3</sub> and Rice Husk Ash on Soil pH at different stages of incubation

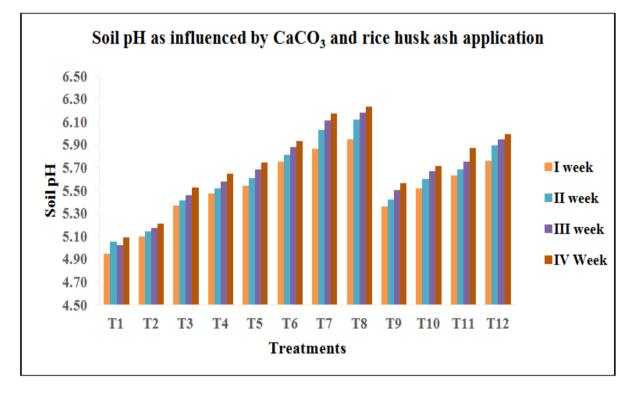
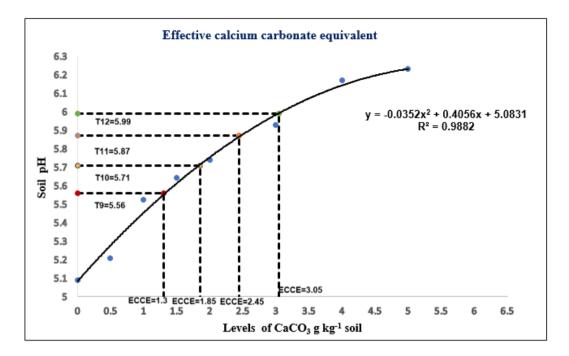


Fig. 1. Soil pH as influenced by levels of CaCO<sub>3</sub> and rice husk ash

# 3.2 Effective Calcium Carbonate Equivalent of Rice Husk Ash

The best-fit regression model to predict ECCE after 4 week of incubation time is quadratic polynomial as shown in Fig. 2. The pH values for RHA levels at 19.4, 29.1, 38.8 and 48.5g kg<sup>-1</sup> soil were 5.56, 5.71, 5.87 and 5.99 corresponding to

ECCE of 1.3, 1.85, 2.45 and 3.05 g  $CaCO_3$  kg<sup>-1</sup> soil. The ECCE values expressed in percentage were 6.70%, 6.35%, 6.31% and 6.28% for RHA levels of 19.4, 29.1, 38.8 and 48.5g kg<sup>-1</sup> soil respectively. The average ECCE of rice husk ash was 6.41 %. The TCCE of rice husk ash used in this experiment was 10.3% and the ECCE of the same is 62.2% of TCCE.



#### Fig. 2. Relationship between CaCO<sub>3</sub> added to soil and soil pH after four weeks of incubation

# 4. CONCLUSION

In the present study, application of rice husk ash @ 48.5 g kg<sup>-1</sup> soil registered comparable soil pH with the equivalent CaCO<sub>3</sub> level of 5.0 g kg<sup>-1</sup> soil. The TCCE (determined by titration method) and ECCE (determined by incubation with the soil) of rice husk ash used in this experiment were 10.3% and 6.41% respectively. Results of the present study confirmed the possibility of using rice husk ash as an amendment for acid soil.

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#### **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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