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Factors affecting maximum moisture up-take of rice husk ash (RHA)

O. J. Ijabo and S. V. Irtwange

Department of Agricultural and Environmental Engineering, University of Agriculture, Makurdi, Nigeria

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Four physical properties of RHA, eleven chemical components and maximum moisture up-take were determined at eight relative humidity levels of 30 to 100%. Particle size, bulk density, particle density, specific gravity, loss on ignition and moisture up-take were determined by gravimetric methods in triplicate. The chemical components, except carbon content, were determined using ICPOES. Single factor ANOVA was used for all determinations that were replicated except for maximum moisture up-take where a 5 x 8 two factor experiment was used. All the properties replicated showed significant difference at $\alpha = 0.05$. That is ash type had effect on all single factor properties while both ash type and relative humidity level affected maximum moisture up-take. All the 23 properties determined were correlated and the results show that maximum moisture up-take in RHA is strongly positively correlated with magnesium oxide and manganese dioxide, fairly correlated with titanium dioxide, but strongly negatively correlated with particle size, alumina, lime and ferric oxide and averagely negatively correlated with phosphorous pentoxide. Fresh dry open heaps of RHA available in rice mills is economical to use as desiccant. Therefore, the cost of sieving RHA from open heaps to reduce the mean particle size should be weighed against direct use in order to obtain maximum benefit when used as a desiccant.

Keywords: Correlation analysis, moisture sorption, ash, desiccant, ICPOES.

INTRODUCTION

RHA is an abundant by-product of rice milling industry which abounds to the level of being a menace if not fully utilized. For example, the potential availability of RHA as reported by Ijabo (2010) is 32, 537, 000 and 195,000 metric tonnes for the world and Nigeria respectively. In Nigeria, which ranks 17th out of the 105 rice producers in the world, RHA visibly abounds as wastes in rice mills all over the country, posing danger to both environment and human health. The global research and use of RHA are mainly directed at its use as an insulator in the steel industry and a pozzolana in the cement and building industry. These R & Ds in RHA utilization indicate properties of RHA which are common to desiccants. For

example Bui (2001) and Essien (2006) reported that the physical property of fineness enhance the pozzolanic activity of cement-RHA composite pastes. Again, Bui (2001) said that pH is a good index of internal pore during reaction.

Similarly, the presence of carbon reported severally (Agbede and Obam, 2008; Reddy and Alvarez, 2006; Basha et al., 2003; Bui, 2001) could be considered. Carbon in the form of activated charcoal is known to be a desiccant so also is the form of silica known as silica gel. In addition oxides like titanium oxide, calcium oxide, magnesium oxide, alkali oxides which are desiccants themselves (Zhang et al., 2007; Staton, 1998; NREL, 2000; GCDT, 2000) are present in RHA.

All these theories could be applied to rate processes like moisture absorption say in air. However, it is not clear how all these attractive properties of RHA would combine

*Corresponding Author E-mail: svirtwange@yahoo.com

when the existing RHA types are used as air dehumidifier. Hence the objective of this study is to determine some physical, chemical and moisture sorption properties of existing five types of RHA with a view to determining the relationship between the properties especially as they relate to maximum moisture up-take (MMU).

MATERIALS AND METHODS

Materials

Five types of RHAs were obtained as follows:

1. The first ash type was obtained from the boiler furnace of OLAM Nigeria PLC, University of Agriculture, Road, Makurdi. This was designated LAM.
2. The second type of ash was collected from an open heap of freshly burnt husks at Aliade rice mills in Benue state, Nigeria. This was designated ALD.
3. The third type of RHA was collected from an open heap of freshly burnt husks at Gboko rice mills in Benue state, Nigeria. This was designated GBK.
4. The 4th type of RHA designated as HAN were obtained by firing dried pre-boiled husks from OLAM in a laboratory furnace set at 600°C for three hours according to Hanfi et al., (1980) method..
5. The 5th type of ash was generated in accordance to Jitcharoen et al., (2009) recommendation for a high specific surface area ash. The method prescribes reflux boiling of the clean husk in 5M HCl for three hours then dried prior to firing in a laboratory furnace at 600°C for another 3 hours. This last type was designated JPC.

Experimental design

A single factor, complete randomized design, experiment was used to test for the significant difference of the mean values for each of the loss on ignition and four physical properties. Each experiment was replicated three times except for specific gravity that was twice. A 5 x 8 two-factor RCBD experiment with the ash types at five levels forming one factor and the relative humidity at eight levels of 30, 40, 50, 60, 70, 80, 90 and 100 percent forming the second factor were used to study the Maximum Moisture Up-take (MMU). The experiments were replicated three times.

Determination of chemical composition using inductively coupled plasma optical emission spectrometry (ICP OES)

Sample decomposition procedure

About 200 mg of the ash powder was weighed into a teflon crucible using a triple-beam balance and dissolved in aqua regia (HCl + HNO₃ + HClO₄ (3:2:1)) after 6 hours of heating the solution to dryness on a hot-plate. After, the crucible containing the sample was allowed to cool, 2 mls of 2M HCl was added and was topped with de-ionized water to about ¾ full and returned to the hotplate to warm for about 15 minutes. For the determination of Silica and Aluminum, 200mg of the ash was weighed in a platinum crucible and fused with Lithium metaborate at a temperature of about 800 °C in a furnace for 10 minutes. It was then brought out and allowed to cool. 5mls of 2M HCl was added to the fused substance and returned to the magnetic stirrer and stirred until it dissolved completely. When crucibles are cool, the content of each crucible was diluted to 100mls and filtered into a flat bottom flask ready to be run on the ICPOES.

Analytical techniques

The samples were analyzed by Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES) at the Department of Geology and Mining of the University of Jos, Nigeria. The instrument was calibrated prior to the introduction of sample by measuring in-house standard and blank solutions. The following trace elements were analyzed; Si, Al, Fe, Ti, Ca, Mg, Na, K, P, S and Mn

The quality of the analysis is controlled through the analysis of samples of known compositions along with the unknowns. The values obtained by the instrument from the reagent blank are automatically subtracted from the raw data before print out. This makes the data free from whatever impurities were inherited from the reagents including distilled and de-ionized water.

The percentage composition of an oxide, POC, e.g. Fe₂O₃, was determined using equation 1.
$$POC (Fe_2O_3) = [(Mol\ wt\ of\ (Fe_2O_3))/(Mol\ wt\ of\ 2Fe)]/[Fe\ in\ ppm/10,000] \dots\dots 1$$

RESULTS AND DISCUSSIONS

The replicate values of bulk density, particle density,

Table 1. A Single Factor ANOVA for Various Parameters of Five Types of RHA

Parameters	Source Variation	of SS	df	MS	F	P-value	F crit
Bulk Density	Ash type	8116.353	4	2029.088	26.96861	2.45E-05	3.47805
	Error	752.389	10	75.2389			
	Total	8868.742	14				
Particle Density	Ash type	5.406787	4	1.351697	3.751157	0.040926	3.47805
	Error	3.603413	10	0.360341			
	Total	9.0102	14				
Particle Size	Ash type	13.48599	4	3.371498	38.83024	4.61E-06	3.47805
	Error	0.868266	10	0.086827			
	Total	14.35426	14				
Specific Gravity	Ash type	0.11794	4	0.029485	4.857496	0.05663	5.192163
	Error	0.03035	5	0.00607			
	Total	0.14829	9				
Loss on Ignition at 1000°C	Ash type	797.3275275	4	199.3319	23.69354	2.46E-06	3.055568
	Error	126.19384	15	8.412923			
	Total	923.5213674	19				

Table 2. ANOVA of Maximum Moisture Up-Take of Five Types of Rice Husk Ash at Different Relative Humidity and at 30°C

Source of Variation	SS	df	MS	F	P-value	F crit
Relative. Humidity	3287.138	7	469.5912	27.15873	1.05E-18	2.126324
Ash Type	3562.79	4	890.6975	51.51335	2.19E-21	2.485883
Interaction	712.9849	28	25.46374	1.472692	0.092355	1.617114
Error	1383.249	80	17.29061			
Total	8946.162	119				

particle size and specific gravity and Loss on Ignition (LOI) of five types of RHA were subjected to analysis of variance as shown in Table 1. Henderson's algorithm, which according to Ijabo (2010) easily shows treatment effects, was preferably used for calculating particle size of the RHA. All the five ANOVA tables indicate that RHA type has effect on each of the five properties. The ANOVA for the replicate values of MMU at the eight relative humidity levels is as in Table 2 indicating that

RHA type and relative humidity both have effect on maximum moisture up-take of RHA.

The other chemical properties apart from LOI were not replicated and are shown in Table 3 together with the mean of other properties that were replicated. To determine how each of these factors relate to MMU, a correlation analysis was carried out and the result shown in Table 4.

In general the physical properties of RHA obtained by

Table 3. Mean* Values of Physical, Chemical and Sorption Properties of Five Types of RHA

S/NO	Class of Property	Parameter	Type of Ash					
			Aliade	Gboko	HAN	JPC	LAM	
1	Physical	Bulk density, kg/m ³	289.7a	312.4a	341.6b	279.1a	283.1a	
2		Particle density, g/cc	1.8050c	3.0354cd	2.6815de	3.6562d	2.6835de	
3		Particle size, mm	0.7823f	1.0330fj	0.5999fk	0.5901gjk	3.0875h	
4		Specific gravity	2.04n	2.21n	2.07n	2.30p	2.13n	
5	Chemical	Total silica	64.89	48.46	71.23	78.45	75.14	
6		Alumina Al ₂ O ₃	0.31	0.22	0.58	0.04	4.61	
7		MgO	11.87	12.32	12.18	12.30	0.80	
8		CaO	0.88	0.56	0.52	0.17	11.46	
9		Fe ₂ O ₃	0.01	0.01	0.00	0.00	0.92	
10		TiO ₂	0.17	0.22	0.11	0.05	0.01	
11		MnO ₂	3.76	3.60	3.59	3.63	0.07	
12		Na ₂ O	1.22	3.07	0.58	0.15	3.66	
13		Potash, K ₂ O	3.03	7.83	0.48	0.13	0.78	
14		P ₂ O ₅	0.77	0.70	0.50	0.47	0.81	
15		LOI	10.43	19.01	9.02	3.50	0.77	
16		Sorption	30% rel. hum.	6.0743a	10.2149ab	6.4604a	10.4322b	1.4201ac
17			40% rel. hum.	7.8252d	12.1474eg	7.2201d	11.7671dg	1.7268f
18			50% rel. hum.	9.7317h	13.4409hj	9.1807h	14.9468h	1.9009hk
19			60% rel. hum.	12.5340m	14.7599m	10.2532	17.8041	1.0434
20	70% rel. hum.		13.7098n	15.1184n	10.7679	18.6541	1.0006	
21	80% rel. hum.		16.8219p	15.7886p	22.7979	23.9924	3.9825	
22	90% rel. hum.		19.7218q	17.8301q	16.1430	22.3041	2.9662	
23	100% rel. hum.		32.3816r	28.6338r	23.9392	27.9028	7.5932	

* Means having the same letter in the same row are not statistically significantly different from each other at $\alpha=0.05$ according to t-Test: Two-Sample Assuming Unequal Variances of Microsoft Excel® (2003)

uncontrolled open heap burning fetched from Aliade and Gboko Rice Mills are not significantly different from each other in terms of bulk density, particle size and specific gravity. This similarity could stem from the use of same husks of the same rice varieties, same uncontrolled open heap burning and same manual parboiling by people of the same culture. Hence, it can be said that mere difference in geographical location has no effect on bulk density, particle size and specific gravity of RHA. Table 3 shows some common chemical components of 5 types of RHA determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES). As expected, Table 3 confirms that silica is the chemical component with the highest percentage in all the five types of RHA with JPC's

ash leading followed by HAN's ash. The chemical component of the RHA that rank second is not common to all the five types of ashes. While ALD, HAN and JPC ashes each has MgO as the second largest chemical component GBK ash has carbon OLAM ash has CaO.

For the third place Aliade, HAN's and JPC's ashes again have carbon in terms of loss on ignition while it is MgO for Gboko ash and calcium oxide for OLAM ash. In terms of carbon content only the two ashes, LAM and JPC, meet the commercial grade standard of having less than 8% as reported by Anon (2003).

In general terms the differences in the chemical composition of RHA types from husk of rice varieties grown in the Lower Benue River basin of Nigeria using

Table 4. Correlation Analysis of RHA Physical, Chemical And Sorption Properties

S/NO	Parameter + units	Bulk Density, kg/m ³	Particle density, g/cc	Particle size, mm	Specific Gravity	Total Silica SiO ₂	Alumina (Al ₂ O ₃)	Magnesium oxide (MgO)	Calcium oxide (CaO)	Ferric Oxide (Fe ₂ O ₃)	Titanium dioxide (TiO ₂)	Manganese dioxide (MnO ₂)
1	Bulk Density	1										
2	Particle density	-0.1077	1									
3	Particle size	-0.3808	-0.1026	1								
4	Specific gravity	-0.3850	0.9264	-0.1007	1							
5	Total silica	-0.3349	0.1942	0.1943	0.0668	1						
6	Alumina (Al ₂ O ₃)	-0.3061	-0.1282	0.9778	-0.1862	0.3481	1					
7	Manganese dioxide	0.3935	0.1064	-0.9844	0.1335	-0.3566	-0.9955	1				
8	Calcium oxide	-0.3806	-0.1232	0.9880	-0.1486	0.3302	0.9961	-0.9995	1			
9	Ferric Oxide	-0.3908	-0.0808	0.9873	-0.1089	0.3448	0.9947	-0.9995	0.9991	1		
10	Titanium dioxide	0.4132	-0.3189	-0.5396	-0.1928	-0.9060	-0.6499	0.6609	-0.6383	-0.6582	1	
11	MnO	0.3659	0.0433	-0.9844	0.0859	-0.3514	-0.9946	0.9971	-0.9963	-0.9988	0.6711	1
12	Sodium oxide	-0.1589	-0.1485	0.8046	-0.0717	-0.4227	0.6869	-0.6897	0.7081	0.7013	0.0350	-0.6969
13	Potash (K ₂ O)	0.1659	-0.1007	-0.1244	0.0748	-0.9811	-0.3030	0.2951	-0.2707	-0.2807	0.8479	0.2886
14	P ₂ O ₅	-0.3571	-0.6268	0.6700	-0.4290	-0.3480	0.5703	-0.5898	0.6085	0.5829	0.1404	-0.5501
15	LOI	0.4998	-0.1521	-0.4818	-0.0730	-0.9481	-0.5997	0.6182	-0.5951	-0.6086	0.9708	0.6109
16	30% rel. hum.	0.1303	0.5374	-0.7960	0.6165	-0.3766	-0.8733	0.8497	-0.8533	-0.8344	0.5159	0.8217
17	40% rel. hum.	0.1076	0.4603	-0.7989	0.5700	-0.4408	-0.8886	0.8620	-0.8635	-0.8481	0.5853	0.8399
18	50% rel. hum.	0.0869	0.4657	-0.8506	0.5613	-0.3146	-0.9155	0.8882	-0.8935	-0.8772	0.5062	0.8692
19	60% rel. hum.	0.0365	0.3786	-0.8813	0.4948	-0.2656	-0.9372	0.9061	-0.9123	-0.8992	0.5005	0.8967
20	70% rel. hum.	0.0251	0.3414	-0.8911	0.4632	-0.2506	-0.9432	0.9114	-0.9178	-0.9061	0.5009	0.9056
21	80% rel. hum.	0.3672	0.3121	-0.9492	0.2348	0.0597	-0.8872	0.8972	-0.9086	-0.8978	0.2725	0.8839
22	90% rel. hum.	0.1022	0.1780	-0.9516	0.2803	-0.2087	-0.9744	0.9521	-0.9578	-0.9529	0.5248	0.9568
23	100% rel. hum.	0.1623	-0.0759	-0.9143	0.0667	-0.4200	-0.9610	0.9440	-0.9412	-0.9481	0.7287	0.9615

Table 4. Continued

S/NO	Parameter + units	Sodium oxide (Na ₂ O)	Potash (K ₂ O)	Phosphorus pent oxide (P ₂ O ₅)	LOI	30% rel. humidity	40% rel. humidity	50% rel. humidity	60% rel. humidity	70% rel. humidity	80% rel. humidity	90% rel. Humidity
11	Manganese dioxide											
12	Sodium oxide	1										
13	Potash (K ₂ O)	0.4830	1									
14	P ₂ O ₅	0.7979	0.3910	1								
15	LOI	0.1253	0.8943	0.0777	1							
16	30% rel. hum.	-0.4723	0.4046	-0.5997	0.5546	1						
17	40% rel. hum.	-0.4404	0.4695	-0.5231	0.6081	0.9943	1					
18	50% rel. hum.	-0.5659	0.3383	-0.6087	0.5117	0.9893	0.9885	1				
19	60% rel. hum.	-0.6302	0.2863	-0.5873	0.4729	0.9597	0.9657	0.9903	1			
20	70% rel. hum.	-0.6511	0.2686	-0.5771	0.4615	0.9445	0.9531	0.9821	0.9987	1		
21	80% rel. hum.	-0.9003	-0.1225	-0.8649	0.2480	0.7645	0.7351	0.8128	0.8321	0.8369	1	
22	90% rel. hum.	-0.7444	0.1949	-0.5754	0.4507	0.8707	0.8849	0.9300	0.9675	0.9775	0.8828	1
23	100% rel. hum.	-0.5963	0.3870	-0.3438	0.6289	0.7843	0.8267	0.8454	0.8915	0.9063	0.7572	0.9529

the ICPOES technique cannot be clearly linked to type of burning or the type of fertilizer applied. However, because of the higher silica content and the smaller particle size of Aliade ash out of the two ashes that are obtained by uncontrolled open heap burning Aliade ash is recommended for further tests involving rate processes. The ANOVA in Table 2 shows that both rice husk ash type and relative humidity of the ambient air have significant effect on the maximum moisture up-take at $\alpha = 0.05$.

From Table 4 it can be observed that magnesium oxide and manganese dioxide have the strongest positive correlation with MMU of RHA at all the relative humidity investigated in this study. The nature of how correlation coefficients vary with relative humidity of the RHA is as shown in Figures 1 and 2 for negative and positive correlations respectively. The correlation coefficients increased steadily with increasing

relative humidity up to 70 % relative humidity and thereafter fluctuate. On the other hand particle size, alumina, calcium oxide and ferric oxide have the strongest negative correlation with MMU. The correlation coefficients in Table 4 are full of surprises. Contrary to the expectation that silica and TiO₂ would be the chemical constituents of RHA to have the strongest positive correlation with and hence the controlling factors of moisture sorption silica has weak negative correlation while TiO₂ has an average positive correlation. The negative correlation exhibited by SiO₂ may be due to the morphology. The silica known to be a desiccant is in the form of a gel while the one correlated with MMU in this study is silica. The reason for TiO₂, a well known desiccant (Jain et al., 1995), having only an average positive correlation with MMU is not obvious, it may be on account of its presence only in trace quantity in the ash compared to other oxides. Alumina too, a

well known desiccant in form of gel, has a very strong negative correlation with moisture sorption probably because of difference in morphology like silica, i.e. alumina gel behaves very differently from alumina in terms of moisture up-take. RHA physical properties like bulk density and particle density both have weak positive correlation with moisture sorption while, as expected, particle size has very strong negative correlation with moisture sorption. The correlation coefficient of particle size with moisture sorption gets larger and larger as the relative humidity increases. Moisture sorption is a rate process just as pozzolanic activity is. Hence, earlier investigators grind the RHA samples for periods ranging from 10 minutes to three hours (Bui, 2001; Anon, 2003) to enhance pozzolanic activity of the ash in block, cement and building industry. In this study involving moisture absorption capacity, the cost of grinding needs to be weighed against the marginal profit derived

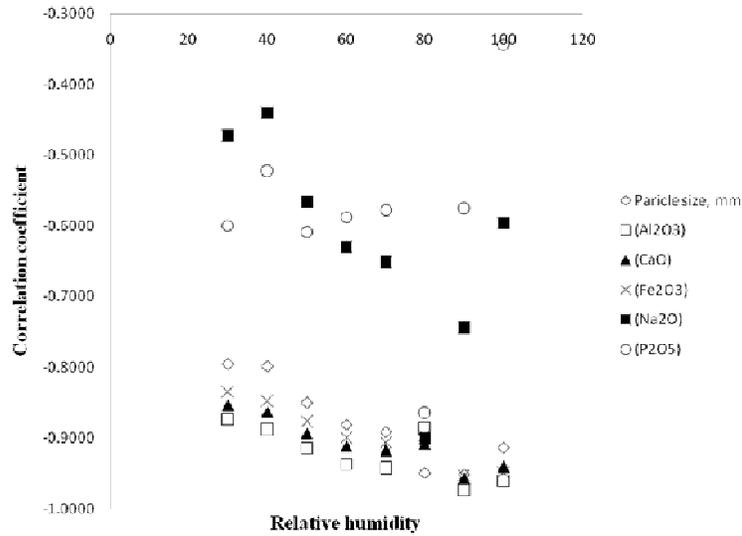


Figure 1. Variation of average and strong negative correlation coefficients of RHA properties.

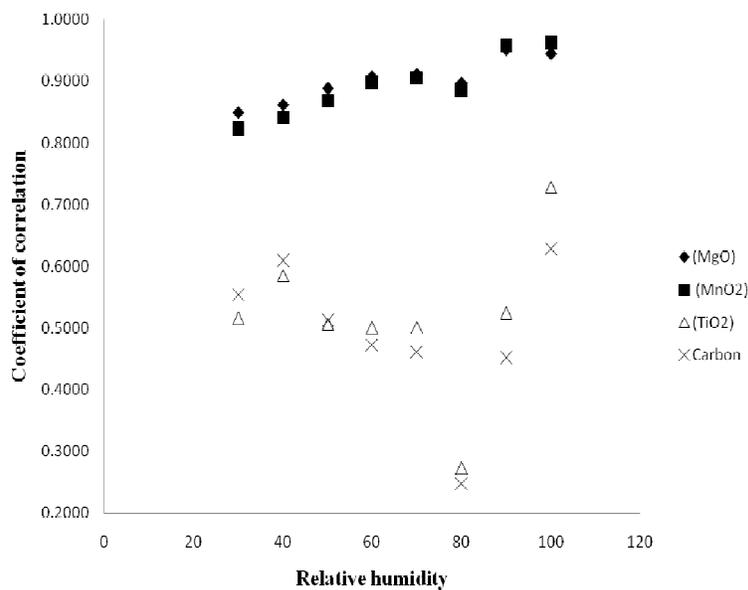


Figure 2. Variation of average and strong positive correlation coefficients of RHA properties.

therein.

Correlation coefficient of physical properties of RHA with their corresponding chemical components is mixed up without any particular direction for example alumina,

lime, ferric oxide and sodium oxide that have a very strong positive correlation with particle size while magnesium oxide and manganese oxide, on the other hand have a strong negative correlation with particle size.

The mean particle size for the five types of rice husk ashes can be arranged in ascending order as follows 0.5901, 0.5999, 0.7823, 1.0330 and 3.0875 mm for JPC, HAN, ALD, GBK and LAM ashes respectively. Table 3 indicates that the particle size of ash from OLAM, 3.0875mm, is significantly larger than all the others in this study subsequently it has the least MMU of all the ashes at all the relative humidity investigated.

CONCLUSION AND RECOMMENDATIONS

Moisture absorption capacity in RHA is strongly positively correlated with magnesium oxide and manganese dioxide, fairly correlated with titanium dioxide, but strongly negatively correlated with particle size, alumina, lime and ferric oxide and averagely negatively correlated with phosphorous pent oxide. Fresh dry open heaps of RHA available in rice mills all over rice producing areas is economical to use as desiccant. Therefore, RHA from open heaps could be sieved to reduce the mean particle size in order to obtain maximum benefit when used as a desiccant, but such marginal benefit should also be weighed against the cost of grinding.

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