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Kath, Aline Hernandez; Islabão, Gláucia Oliveira;
Vahl, Ledemar Carlos; Teixeira, Juliana Brito da Silva
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Reaction rate and residual effect of rice husk ash in soil acidity parameters¹

Aline Hernandez Kath^{2*}, Gláucia Oliveira Islabão³, Ledemar Carlos Vahl², Juliana Brito da Silva Teixeira²

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ABSTRACT

The rice husk ash has been applied in agricultural land, with potential of replace limestone and, supply phosphorus and potassium. However, its residual effect in soil is still unknown. This investigation aimed to evaluate the reaction rate and residual effect of rice husk ash in soils acidity parameters. A field experiment was conducted with five treatments: four rice husk ash dosages 0, 30, 60 and 120 t ha⁻¹ and one treatment with recommended soil lime and fertilizer (dolomitic limestone to reach pH 6, 150 kg ha⁻¹ P₂O₅ as single superphosphate and 80 kg ha⁻¹ K₂O as potassium chloride) where five soil samples. Soil samples were collected in the layers 0.00–0.10 m and 0.10–0.20 m at 15, 211, 400, 517 and 804 days after ash incorporation. Chemical attributes were determined: soil pH (pH), soil base, exchangeable cation values (Ca, Mg, K and Na) and cation exchange capacity (CEC) at pH 7. Results showed that reaction rate of rice husk ash is faster when compared to liming. As greater was rice husk ash dosage applied in soil, higher is the residual effect in pH. As corrective of soil acidity, the residual effect of rice husk ash is just the required time to occur the natural process of reacidification and leaching of basic cations, about 33 months for soils and weather conditions similar to this work.

Keywords: waste; fertility; time; agriculture.

RESUMO

Velocidade de reação e efeito residual da cinza de casca de arroz nos parâmetros de acidez do solo

A cinza de casca de arroz apresenta como uma de suas principais utilizações a incorporação na agricultura, com potencial para substituir a calagem e fornecer cálcio e potássio. Entretanto seu efeito residual no solo ainda é desconhecido. Este trabalho teve como objetivo avaliar a velocidade de reação e o efeito residual da cinza de casca de arroz nos parâmetros de acidez do solo. Para tal, foi realizado um experimento de campo constituído por cinco tratamentos: quatro doses de cinza de casca de arroz 0, 30, 60 e 120 t ha⁻¹ e um tratamento com adubação e calagem recomendada (calcário dolomítico para atingir pH 6, 150 kg ha⁻¹ P₂O₅ como superfosfato simples e 80 kg ha⁻¹ K₂O como cloreto de potássio) onde foram realizadas cinco amostragens de solo, nas camadas de 0.00-0.10 m e 0.10-0.20 m, aos 15, 211, 400, 517 e 804 dias após a incorporação da cinza. Os atributos químicos analisados foram: pH em água, porcentagem de saturação de bases, teores de cátions trocáveis (Ca, Mg, K e Na) e capacidade de troca de cátions (CTC) a pH 7. Os resultados indicaram que, a velocidade de reação da CCA é muito rápida quando comparada ao calcário. Quanto maior for a dose de cinza de casca de arroz aplicada ao solo, maior é o efeito residual no pH. E, como corretivo de acidez, o efeito residual da cinza de casca de arroz resume-se ao tempo necessário para ocorrer o processo natural de reacidificação e lixiviação dos cátions básicos, cerca de 33 meses para solos e condições climáticas similares ao deste trabalho.

Palavras-chave: resíduo; fertilidade; tempo; agricultura.

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² Universidade Federal de Pelotas, Departamento de Solos, Capão do Leão, Rio Grande do Sul, Brazil. alinecou@gmail.com; ledovahl@hotmail.com; julianabrito@gmail.com

³ Instituto Federal de Farroupilha, Departamento de Química, Alegrete, Rio Grande do Sul, Brazil.

*Corresponding author: alinecou@gmail.com

INTRODUCTION

Rice husk ash (RHA) is a by-product from the burning of rice husk to energy production. It is an alkali product and serves as corrective of soil acidity. Islabão *et al.* (2014) evaluated the RHA quality as corrective of soil acidity and concluded that it has a very low neutralizing value (around 1%). Although it reacts much faster than conventional limestone, it has a low effective calcium carbonate equivalent (around 3%). Nevertheless, as the doses used for farmers in their small farms are about dozens of tons per hectare, the use of this residue may replace limestone with the advantage of also supplying phosphorus and potassium (Islabão, 2013).

The liming action time to correct soil acidity is long and reapplication criteria are well established. The limestone residual effect is equivalent to the time period in which acidity and toxic aluminum are controlled (Novais & Mello, 2007). About ashes this issue is unknown but has fundamental importance for its use as liming replacement.

According to Natale & Coutinho (1994), residual effect depends on several factors: liming, dosage requirement, type of soil, fertilization, cropping system, among others. However, an important factor is the limestone reactivity: a higher reactivity results in a lower residual effect, this is, the faster the limestone action, the shorter the time of its effects and vice versa.

The loss of effects in the soil over time - as happens with the limestone - are due to reacting with acidic substances, combined with the loss due to erosion, leaching and removal by crops (Cruz, 1977) probably also occurs with the RHA.

The qualitative composition of the bases present in RHA is unknown but it may be similar to other ashes from plant biomass. According to Demeyer *et al.* (2001) several studies have shown that wood ash reacts in soil faster than limestone, resulting in a greater pH increases. However in a short period of time hydroxides and K and Na carbonates, which are more soluble and, do not persist in the soil for a long time. Agricultural limestone solubilization is slow, once it is composed by Ca and Mg carbonates which have low solubility. In addition, the common limestones have particles in enough size to be solubilized on long-term, which increases the residual effect (Raij, 2011).

The objectives of this study were to evaluate the reaction rate and residual effect of rice husk ash in soil acidity parameters from a Typic Hapludult.

MATERIAL AND METHODS

It was carried out a field experiment at the Agricultural Center of Palma, Universidade Federal de Pelotas (UFPeL),

of the town of Capão do Leão – Rio Grande do Sul/RS (coordinates E357.860 N6.480.490; UTM zone 22; datum WGS84). The soil of the experimental is a Typic Hapludult (Severo, 1999) with a sandy loam texture.

The RHA dosages were equivalent to 0, 30, 60 and 120 t ha⁻¹ and an additional treatment followed the recommendation of the Comissão de Química e Fertilidade do Solo – CQFSRS/SC (2004) (recommended fertilization – RF) from work of Islabão *et al.* (2014). The RHA composition is on Table 2.

Both RHA and RF were incorporated into the surface layer (0.00-0.10 m) with a rotary tiller. The experimental plots had 6 x 4 m and were arranged in a randomized block design with four replications. The ash produced through combustion of rice husk in a fluidized bed combustor was provided by a company from Pelotas – RS, Brazil. Fluidized bed technology is capable of producing amorphous rice husk ash with very low carbon content in a very rapid reaction time (Angel *et al.*, 2009). The RF consisted of lime requirement to reach pH 6.0. The calculated dose (SMP method) for each block were 0; 0.8; 1.1 and 2.7 t ha⁻¹ of dolomitic limestone with ECC = 76.2%. A dose of 150 kg ha⁻¹ P₂O₅ as single superphosphate and 80 kg ha⁻¹ K₂O as potassium chloride were applied in the RF treatment.

Two crops (black oat and common beans) were sown in April and November 2010, respectively. The vegetable residue from crops was kept as soil cover after the harvest and, after common bean crop, the area was revegetated with native grassland.

The five soil samples were collected on the following dates: 1ª) 15 days after application (daa) of RHA; 30/04/2010; 2ª) 211 daa: 12/11/2010; 3ª) 400 daa: 20/05/2011; 4ª) 517 daa: 14/09/2011 and 5ª) 804 daa: 27/06/2012.

The collects were made in two independent samples in each layer (0.00- 0.10 m and 0.10-0.20 m). Each sample consisted of six sub-samples per plot collected with a soil column cylinder auger. The samples were dried at 60 °C to constant weight, loosened by hand with a clod breaker and sieved (< 2 mm).

The chemical and physical characterization of the soil (Table 1) was performed at laboratory of Soil Science Department/UFPeL. Calcium (Ca), Magnesium (Mg), Potassium (K), Sodium (Na) and pH_{H2O} were determined through analytical methods described by Tedesco *et al.* (1995). The cation exchange capacity (CEC) at pH 7 and percent base saturation (BS) were calculated from the analyzed variables.

Rainfall data from experimental period, obtained in the website Agro-Meteorological Station of Pelotas (Embrapa, 2013) located about 7 km from experimental area is shown in Figure 1. The Agrometeorological Station works through the agreement Embrapa/UFPeL/INMET.

A Two Way ANOVA Repeated Measures analysis of variance was performed to analyze the data of soil chemical properties because it allows evaluating the variables effects as well as the interaction between them. It were also run a regression analysis between variables and time whose significance was evaluated by F test. Both analyzes were made using the computer program SigmaPlot® (2008).

From adjusted equations through regression analysis were calculated total time (tt) and half-life ($t_{1/2}$) of treatment effects in chemical attributes.

RESULTS AND DISCUSSION

Since the focus of this work are the residual effect and reaction rate of RHA, emphasis was given in time effect on soil attributes. In attributes affected by treatments, the effects were more accentuated in layer 0.00-0.10 m, where RHA was incorporated, but also occurred in layer 0.10-0.20 m. In all variables studied, analyzes of time effects in each layer individually not added any improvement in the interpretation with regard to average data from two layers

Table 1: Chemical properties and particle size of experiment soil in the layers 0.00-0.10 m and 0.10-0.20 m

pH H ₂ O	Exchange cations										
	OC	P	K ⁺	Ca ⁺²	Mg ⁺²	Na ⁺	CEC _{pH7.0}	BS	Clay	Silt	Sand
	g dm ⁻³	mg dm ⁻³	mmol _c dm ⁻³				%	g kg ⁻¹			
							0.00-0.10 m				
5.54	11.30	5.8	1.64	23.60	10.51	0.80	76.19	49.17	169.3	224.4	606.3
							0.10-0.20 m				
5.23	9.85	3.6	1.21	27.4	11.30	0.82	70.56	59.12	192.9	220.0	586.9

OC = organic carbon, BS = percent base saturation; CEC_{pH7.0} = CEC at a pH 7.0

Source: Adapted of Islabão *et al.* (2014)

Table 2: Chemical composition of the rice husk ash used in the experiment

pH (H ₂ O)	NV	Si	K	Na	Ca	Mg	Al
	%	g kg ⁻¹					
8.6	0.91	410.5	11.35	6.83	6.02	4.2	0.23
C	N	P	S	Cu	Fe	Mn	Zn
g kg ⁻¹				mg kg ⁻¹			
72.3	0.98	2.64	3.52	7.5	480	1278	34

NV= Neutralizing value

Source: Islabão *et al.* (2014)

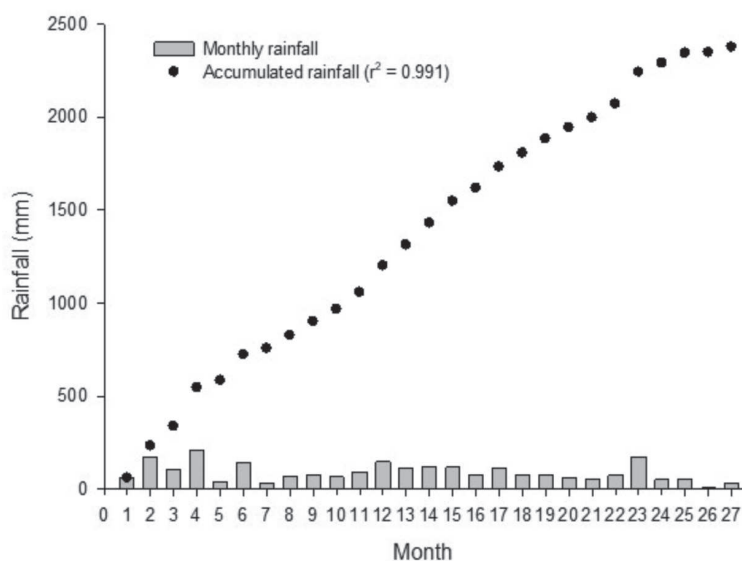


Figure 1: Monthly (bar) and accumulated (dotted) rainfall in the experimental period of 15/04/2010 to 27/06/2012.

representing the superficial soil layer 0.00-0.20 m. Then it was decided to develop the whole interpretation with superficial layer data (0.00-0.20 m).

The effects of dose and time after RHA application in the soil exchangeable bases contents (Ca, Mg, K and Na) were statistically significant at the probability level of 0.1% or less, except for the effect of dose in Ca, which was significant at 6.2% (Table 3). The interaction between dose and time was significant to the monovalent cations (K and Na), but not to the divalent cations (Ca and Mg). This means that variations in K and Na contents with the time depended on the dose of RHA applied, but the variations of Ca and Mg with the time did not depend on the dose of RHA.

As soon as RHA was applied, contents of Ca, Mg, K and Na increased and then, decreased with the time in all doses, including zero dose, except for Na that it remained stable over time in the treatment without RHA (Figure 2). Both the initial increase with RHA application and the reduction model over time dependent on each cation. The increases in cations contents, with RHA application in the beginning of the experiment were presented and discussed by Islabão (2013). However, it should be noted the variation in the magnitude of these increases among cations, as can be seen at the data corresponding to sampling done at 15 daa in Figure 2. Comparing the extreme RHA applications, zero and 120 t ha⁻¹, Ca increased about 20%; Mg contents doubled, K contents quadrupled and Na increased 16 times with this treatment.

To evaluate the time effect, regression analysis was used between each cation content and time after applying within each RHA dose, even in cases where the dose x time interaction was not significant, as a matter of uniformity in data processing. Decreasing in levels with the time was linear to divalent cations, Ca and Mg, and exponential to monovalent cations, K and Na (Figure 2).

The contents of exchangeable Ca tended to remain stable during the first three sampling times (until 400 days) and decreased sharply in the last two samplings, in all RHA applications (Figure 2a). Despite this, the linear regression adjustment between Ca contents and the time was significant. Decreased Ca rates with the time (slope of the fitted straight) varied very little between treatments

(0.00739 mmol_c dm⁻³ day⁻¹ at the zero RHA treatment to 0.00836 mmol_c dm⁻³ day⁻¹ at the 120 t ha⁻¹ RHA treatment). As there was not significant interaction between time and RHA application (Table 3), these differences in slope of straight between the RHA treatments were not large enough to be statistically significant.

Magnesium behavior was similar to Ca. Its content has declined linearly with the time in all RHA doses. Although dose x time interaction also has not been statistically significant, the decrease of Mg content with time (slope of the fitted straight) varies more consistently with RHA application. The Mg content decreases faster with the time in highest RHA applications, varying the rate of decrease of 0.00313 mmol_c dm⁻³ day⁻¹ at the zero RHA treatment to 0.00732 mmol_c dm⁻³ day⁻¹ at the 120 t ha⁻¹ RHA treatment (Figure 2b). Similarly to the Ca, these variations in the rate of decrease in the Mg contents between RHA applications were not large enough to reach statistical significance.

The contents of exchangeable K and Na have declined exponentially with the time but the curvature equations was sharper at Na than K and in higher RHA than lower dose for both cations. (Figure 2c and 2d).

In principle, the process responsible for decreasing contents of exchangeable cations with time, was leaching to deeper soil layers (beyond 0.20 m). During the experiment period (804 days) the rainfall was relatively uniform, and their accumulation almost linear with the time (Figure 1). The exception were the first days after application of RHA, in which occurred heavy rain (55.5 mm between the third and the fifth day after application of RHA), probably causing high leaching of Na, already checked in the first sampling at 15 daa, as discussed by Islabão (2013).

The comparison between cations in terms of losses model and magnitude versus time can be more appropriately performed using the relative contents of each cation in relation to initial content of each one. Figure 3 shows the relative contents of the four cations versus time relative to 120 t ha⁻¹ RHA treatment according to adjusted equations in Figure 2. It is clear that the magnitude of relative loss with the time occurs in the order: Na > K > Mg > Ca. This is the reverse order

Table 3: Significance levels obtained from analysis of variance to repeated measures (Two-Way Repeated Measures ANOVA) of exchangeable basic cations Ca, Mg, K and Na, percent base saturation (BS) and pH

Factor	Ca	Mg	K	Na	BS	pH (H ₂ O)
	P ⁽¹⁾					
Dose	0.062	< 0.001	< 0.001	< 0.001	< 0.001	0.002
Time	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Dose x Time	0.813	0.428	< 0.001	< 0.001	0.021	< 0.001

⁽¹⁾P: Probability due to chance.

of strength of adsorption by soil (Sposito, 1989) and, therefore, is coherent with what would be expected: as stronger the adsorption by the soil, lesser is the lost by leaching.

The difference between cations on the losses model over time, linear for the divalent cations and exponential to monovalent, may just be an experimental artifact. There is no theoretical justification to the loss model over time be different between mono and divalent cations. Besides, Markewitz *et al.* (2006) and Werle *et al.* (2008) observed that as K as Ca and Mg had exponential losses, increasing the loss rate depending on the initial content of each cation. As discussed before, Mg loss rate tended to increase with RHA dose (slope of the fitted straight in Figure 2b), with initial Mg content, but the relative rates were small at all doses. Calcium loss rates were even lower. It is possible, therefore, that the exponential character of relationship has not manifested more clearly for the divalent cations

due to small magnitude of losses of Ca and Mg (compared to K and Na), resulting in a linear relationship between cation content and the time.

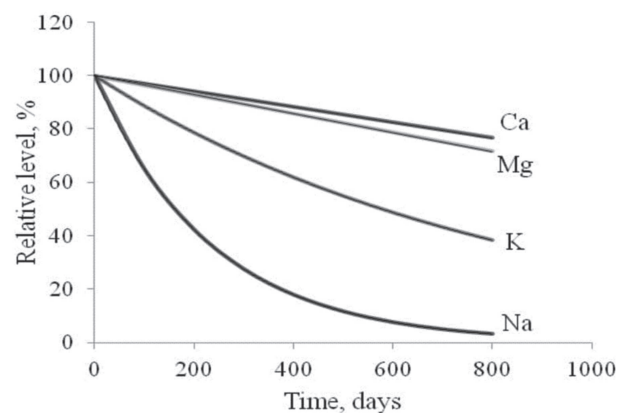


Figure 3: Relatives contents (%) of exchangeable cations over time (days) at a 120 t ha⁻¹ rice husk ash treatment.

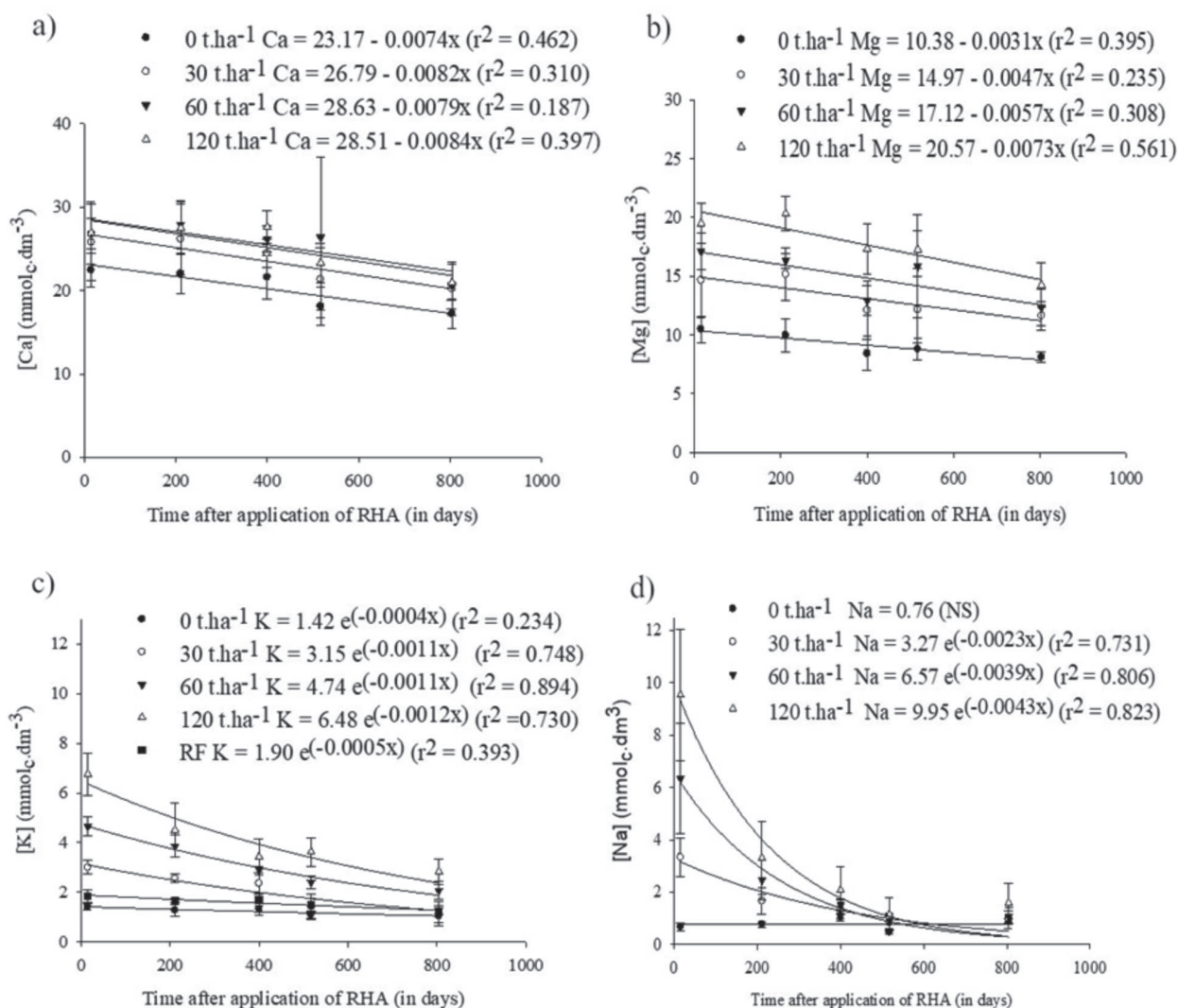


Figure 2: Contents of exchangeable cations over time after application of rice husk ash (RHA) and recommended fertilization (RF) at the doses indicated by adjusted equations in layer 0.00 – 0.20 m of a Typic Hapludult.

On the other hand, decreasing of Ca, Mg and K contents over time, even at zero RHA treatment, shows that concentrations of these cations were above what is natural for this soil due to fertilization and liming performed for crops. The magnitude of these losses agrees with the observed in the others treatments: are smaller in the zero RHA than in plots with RHA because the initial contents were smaller.

The CEC at a pH 7 did not change. The time effect on percent base saturation (BS) is the sum of the effects on exchangeable basic cations Ca, Mg, K and Na. The BS and the pH decreased over time more consistent than was observed for the cations individually. The coefficients of determination of linear equations adjusted to the data were all above of 0.8 (Figure 4). The slope of straight increased by RHA application in these two parameters, which means that the rate of decrease with the time has increased with the initial value to both BS and pH. In other words, as greater the initial value (highest RHA application) the higher was the rate of decrease with time.

Figure 5 (rate of decrease calculated from slope equations of the curve per year) shows the relationship between BS rate of decrease along with the initial BS, and pH rate of decrease. Apparently pH and soils base saturation tend to return to originals values which are in the soil in their natural state. Theoretically these values correspond those that would be observed when the rate of decrease is zero in Figure 5. These values (pH = 4.96 and BS = 29%) are very close to the average values observed by Nachtigall & Vahl (1989) in the average of seven Typic Hapludult from region in their natural state, without cultivation.

The decrease of the BS and pH over time shows the natural process of soil reacidification after acidity correction. In areas where rain exceeds evapotranspiration,

soil acidification is a natural process (Bolan *et al.*, 2003). The acidification phenomenon may involve several processes, but the main acidity source for soil is the CO₂ derived from respiration by plant roots and microorganisms, which dissolves in water to form carbonic acid (Schlesinger & Andrews, 2000).

When CO₂ dissolves in water, it forms carbonic acid that dissociates to produce ions HCO₃⁻ and H⁺ according to reaction: CO₂ + H₂O <=> HCO₃⁻ + H⁺ (Sposito, 1989). Under normal CO₂ concentration in the air (0.03%), pH of rain water is about 5.6 (Bolan *et al.*, 2003). In soil air the CO₂ concentration is even higher and pH of solution could still be less than 5.6 (Sposito, 1989). The most important fact is that over time, soil receives rain water with a concentration of at least 10^{-5.6} mol L⁻¹ of H⁺ which is added to soil. The ions H⁺ have high affinity by pH-dependent charges of soil colloids, displacing base cations from these charges. This decreases the CEC and the base cations that are released into soil solution are subject to leaching by percolating water.

In treatment with liming (RF), the pH reached value close to 6 (5.8) only in the second sampling, at 211 daa, which was expected because limestone reaction is sufficiently slow to not exercising their corrective effect already in the first sampling (15 daa). The pH value ranged between 5.5 and 5.8 over time but without defined trend and an average 5.6. According to Zancanaro (1996) there are seasonal variation of soil pH of 0.3 at 0.5 units with the time in soil without liming and, in soils with low LR (lime requirement), the liming effect can be masked by this seasonal variation. In this treatment BS showed decreasing trend with the time, but this decrease was not statistically significant. Therefore, liming effect remained over the 804 days of the experiment.

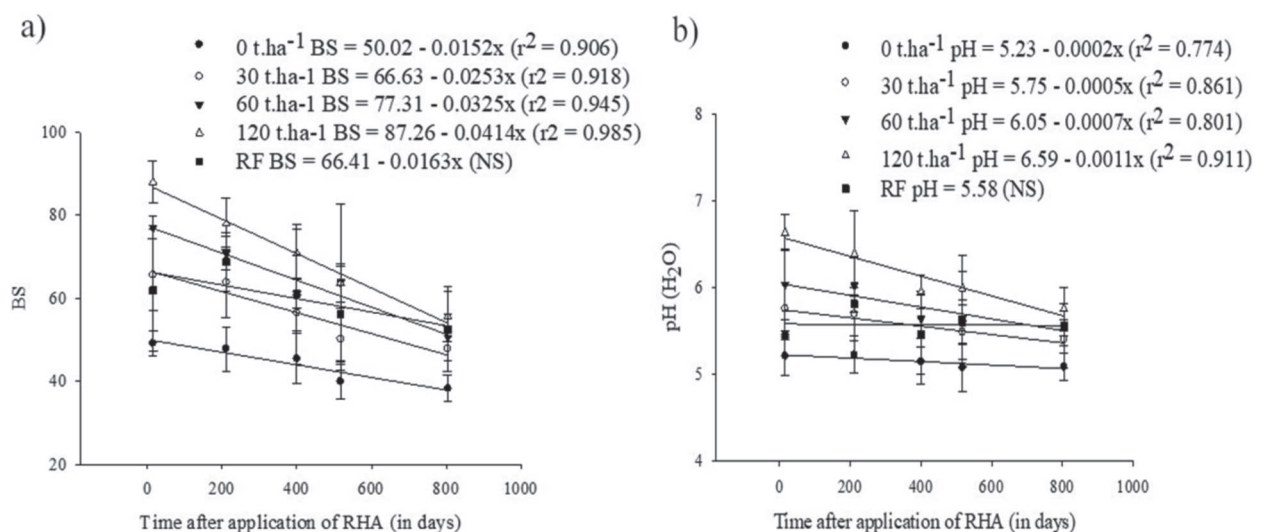


Figure 4: Time effect of application of rice husk ash (RHA) and recommended fertilization (RF) on a) percent base saturation (BS) and b) pH (H₂O) in layer 0.00 – 0.20 m of a Typic Hapludult.

Comparing the RHA effect with liming effect, there are two important aspects: the initial reaction rate and the residence time of effect in acidity parameters, BS and pH. While the RHA effect was already full in the first sampling (15 daa) and thenceforth has begun to decline, the liming effect has reached the peak effect at 211 daa and remained until the end of sampling period (Figure 4).

Considering the residual effect of RHA as the residence time of effect measurable in the analyzed variables, it was estimated the required time to the basic cations contents. The BS and pH return to values obtained in the beginning of experiment in the treatment zero, representing soil in their initial state. For this estimate were used the equations listed in Figures 2 and 4. The estimated times are listed in Table 4. Just Ca and Na had residence time smaller than 804 days after application of RHA. To the other variables, the estimated time extrapolated the 804 days, mainly at the highest RHA application. The $t_{1/2}$ of all variables, in all three RHA doses, were smaller than 804 days (Table 4). Obviously, in the variables that the relationship with the time is linear the $t_{1/2}$ is half the time to the total extinction of the effect and in variables which the relationship is exponential (K and Na) the $t_{1/2}$ was lower than total time.

Except for Na, in other variables the $t_{1/2}$ increased by RHA application, but not in direct proportion: a fourfold increase in RHA application (30 to 120 t ha⁻¹) increased the $t_{1/2}$ just between 18 and 44%, depending of variable. On the other hand, the ascending order of $t_{1/2}$ between four basic cations was Na < Ca < K < Mg (Table 4). This is not exactly the reverse order of these cations due to rate of decrease by leaching that, in principle, it would be logical: Ca, which had the lowest rate of decrease, should have the greatest $t_{1/2}$; the other three cations results are in agreement with their rate of decrease. In the case of Ca, increased from their initial content by RHA application was very small, just 20% compared to the soil content. So, although the Ca rate of decrease was the lowest between the cations it was enough to lose faster the small amount of Ca that RHA added to the soil.

The $t_{1/2}$ of BS is the weighted median of $t_{1/2}$ from absolute contents of basic cations. So, like the $t_{1/2}$ of each cation increased by RHA application, the $t_{1/2}$ of BS also increased. As the pH is related to the base saturation, it increased accordingly (Table 4). However, there is inconsistency between pH and BS of $t_{1/2}$ values: they should be, at least, very close each other. It's possible that

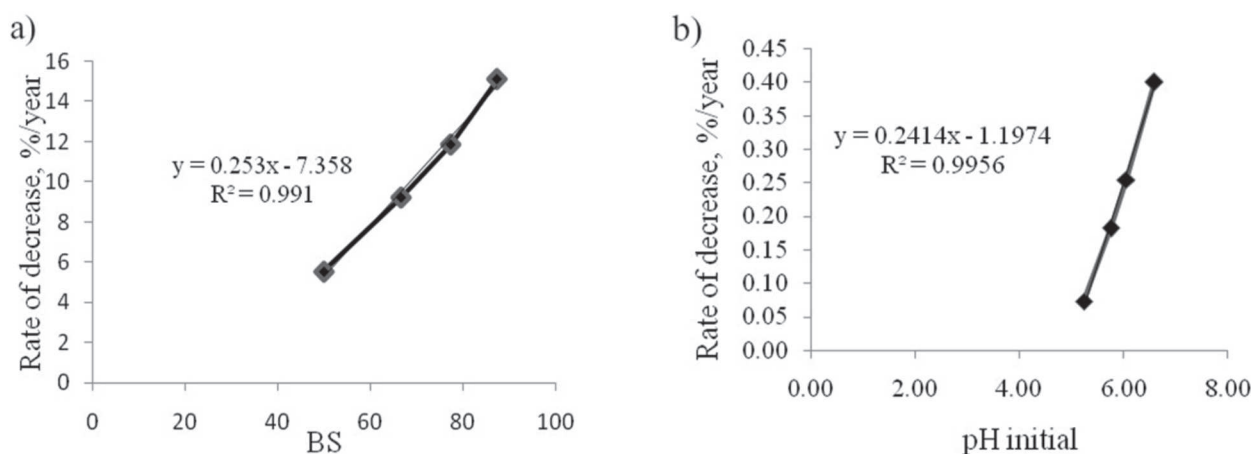


Figure 5: Rate of decrease (year) to a) percent base saturation (BS) and b) pH (H₂O) in relation to their respective initial values in a Typical Hapludult in layer 0.00 – 0.20m.

Table 4: Total time (tt) and half-life ($t_{1/2}$) estimated to contents of exchangeable cations, base saturation (BS) and pH values returned to initials values in soil

Dose	Ca		Mg		K		Na		BS		pH	
	tt	$t_{1/2}$	tt	$t_{1/2}$	tt	$t_{1/2}$	tt	$t_{1/2}$	tt	$t_{1/2}$	tt	$t_{1/2}$
t ha ⁻¹	Days											
30	441	220	977	488	724	292	634	215	656	328	1040	520
60	691	346	1182	591	1095	392	553	151	840	420	1171	586
120	636	318	1396	698	1265	412	598	145	899	450	1236	618
RF ⁽¹⁾	-	-	-	-	582	270	-	-	-	-	-	-
53.66 ⁽²⁾	-	-	-	-	-	-	-	-	809	404	1149	574

⁽¹⁾Treatment with limed and fertilized recommended by the CQFSRS/SC (2004).

⁽²⁾RHA dose estimated to correct the initial soil pH to 6.

this inconsistency is just a consequence of accumulated experimental errors, because the $t_{1/2}$ were estimated from adjusted equations between the variables and the time which variation rates were numerically small and, therefore, the experimental error becomes large proportionally. Regardless of this apparent inconsistency, the RHA effect as acidity corrective is almost immediate, as discussed by Islabão *et al.* (2014) and starts to decrease just after their application into the soil. In the necessary RHA dosage to increase the initial pH to 6, estimated in 53.66 t ha⁻¹, the $t_{1/2}$ was 574 days to the pH and 404 days to the BS (Table 4). Even with this discrepancy of $t_{1/2}$ between pH and BS, is a much smaller duration time than that observed to liming effect, in RF treatment, which there was not significant variation in the pH and in the BS during 804 days of experiment. The projection for the total time (tt) was 809 days for BS and 1149 days for pH (Table 4). On average, this means about 33 months that can be considered as the time of the residual effect of RHA as corrective of acidity for the soils and weather conditions studied.

CONCLUSIONS

Reaction rate of RHA is faster when compared to liming, because all bases contained in it are dissolved immediately after their incorporation into the soil.

The residual effect of RHA on exchangeable Ca, Mg and K contents, on the base saturation and in the pH increased with increasing RHA applied to the soil.

As corrective of soil acidity, the residual effect of RHA comes down to the time needed to occurs the natural process of reacidification and leaching of basic cations, about 33 months for soils and weather conditions similar to this work.

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