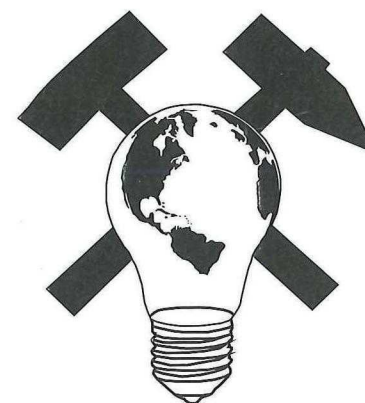


JAARBOEK

**VAN DE MIJNBOUWKUNDIGE
VEREENIGING TE DELFT**



**77^e Editie
2015-2016**

**Shifting Sources
-
Long Term Supplies**

Rice husk ash with high carbon content proves favourable for soil stabilization

door: Vinh Pham e.a.

Vinh Pham^{1*}, Wouter RL van der Star², Guang Ye³, Leon A. van Paas-sen¹

¹ Delft University of Technology, Department of Geosciences and Engineering

² Deltares, Subsurface and Groundwater Systems

³ Delft University of Technology, Department of Structural Engineering

Abstract

Rice husk ash is a promising pozzolanic material produced from rice husk burning and has significant potential as a sustainable replacement for cement in construction and ground improvement applications. In this study the effect of burning conditions on the ash reactivity and its potential for soil stabilization applications have been investigated. Three different burning procedures were applied: 1) controlled burning at 500°C followed by rapid cooling, which was according to literature considered to produce the highest reactivity, 2) burning at 700°C with slow heating and cooling, which was expected to result in lower reactivity due to the crystallization of the silica and 3) uncontrolled burning in open-fire. The third procedure unexpectedly produced the most reactive ash, even though it contained less silica

and still some carbon. Adding rice husk ash and lime to wet clay significantly increased the strength of the clay after curing. Again, the carbon-containing ash showed the largest strength improvement. Incomplete combustion seems therefore preferable for soil stabilization applications with rice husk ash.

Keywords: soil stabilization, clay, rice husks, burning conditions, pozzolanic material.

1. Introduction

Rice husks (RH) are the hard coatings protecting rice grains during the growing season. Combustion of rice husks produces rice husk ash (RHA) which is a promising pozzolanic material. It can be used as a sustainable alternative for cement in building materials, like concrete (UNIDO 1984; Cook 1986; Malhotra and Mehta 1996; Van Tuan et al. 2011) or in soil improvement applications (Basha et al. 2005; Muntohar 2006; Muntohar et al. 2013). In general, silica can accumulate in local parts of plants as a result of evaporation (Handreck and Jones 1968). In the case of rice plants silica accumulates in the rice husks forming the protective hard coating. Krishnarao and Godkhindi (1992) showed that the silica in rice husk was amorphous and bonded

to organic substances. Burning RH removes the organic components and increases the proportion of silica from 20% in RH to about 90% in RHA (Rao et al. 1989). The amorphous silica in RHA is a pozzolanic material, which means that it is able to react with different alkaline compounds. The pozzolanic reaction is expected to occur when RHA is mixed with an alkaline compound such as calcium hydroxide under moist conditions to form compounds with cementing properties (Malhotra and Mehta, 1996). Among the various pozzolanic materials, RHA is categorized in the group of the most reactive agents (Agarwal 2006; Walker and Pavía 2011).

The reactivity of RHA is attributed to its high amorphous silica content and small grain size, which are controlled by the burning conditions applied to the original RH and grinding energy applied to the ash after burning. To completely remove the organic content, rice husks require 12 hours combustion at 500°C (UNIDO 1984; Nair et al. 2008). The combustion time can be shortened by increasing the temperature, but too high temperatures can cause the amorphous silica to crystallize, which lowers the reactivity (Rao et al. 1989). The crystallization of silica is sensitive to the burning and cooling duration and temperature. In some cases the formation of quartz was observed at burning temperatures above 600°C. According to the overview by UNIDO (1984), silica remains amorphous for

prolonged time at a temperature of 500°C. Above this temperature the holding time at which crystallized silica was detected varied corresponding to the temperature and local conditions, e.g. less than one minute at 680°C recorded by Mehta (1979), or less than one hour at 900°C recorded by Yeoh et al. (1979). The general temperature range reported in later studies to produce amorphous silica is between 500°C and 800°C; For example Behak and Núñez (2013) recently stated that the optimum burning temperature to produce highly reactive ash was between 650 and 800°C and indicated that above 900°C the ash started to crystallize and became less reactive. Grinding the ash improves the reactivity because it reduces the grain size and increases the reactive surface area, hence resulting in a higher reaction rate with calcium hydroxide. For example, Cordeiro et al. (2011) showed that reducing the mean diameter from 200 µm to 20 µm by grinding, lead to an increase of the pozzolanic activity by a factor of two. However, the crystalline state of silica affects the reactivity of the siliceous material to a much greater extent than the particle size (Walker and Pavía 2011). Evaluating the required energy for both burning and grinding, Muthadhi and Kothandaraman (2010) proposed that burning for 2 hours at 500°C is the optimum condition to produce reactive RHA.

Owing to its pozzolanic activity, RHA is used as an additive in combination

with lime (calcium hydroxide), for soil improvement and has shown promising results. For example, A. Seco et al. (2011) demonstrated that mixing lime and RHA with an expansive soil from Tudela, Spain resulted significant improvement of strength and reduction of the swelling capacity. Some researchers intended to produce high quality ash in their experiments (A.J. Choobasti et al. 2010), while others used uncontrolled burning conditions, either by collecting the ash from energy plants (Muntohar 2002; A. Seco et al. 2011) or just by burning the husks directly at the rice mills in open fire (F. Haji Ali et al. 1992) or by using an incinerator (Basha et al. 2005; Muntohar 2006). Interestingly in all the cases, the soil was considerably improved. Behak and Núñez (2013) looked at the effect of burning conditions and showed that RH burned at 650 and 800°C resulted in much higher strength than RHA burned at uncontrolled conditions at the local rice parboiling plant when mixed with lime and sandy soil. The various literature sources seem to be ambiguous about the optimum burning conditions to produce high quality RHA ash for soil stabilization applications as they either did not analyse the reactivity of the ash or they used different grinding procedures, different mixing ratios or different soil types.

In this study, the effect of burning conditions on the reactivity of the ash and its potential for ground improvement applications in clayey soils was investi-

gated. Three different burning conditions were applied to produce the RHA. The properties of three types of ash were analyzed and their potential for soil stabilization was evaluated by determining the geotechnical properties (consistency limits, compressibility and undrained shear strength) of various mixtures of clayey soil, RHA, lime and water using standard laboratory experiments.

2. Materials and methods

2.1. Materials

2.1.1. Soil

The soil used for the experiments was a commercially available river clay (K-10000, Ve-Ka, Netherlands). According to ISO 14688 (CEN 2002) the soil is classified as silty clay with high plasticity. The clay was delivered with a water content of approximately 35%. It was then mixed with tap water to obtain the desired liquidity at a water content of 48%, corresponding with Liquidity Index of 0.76.

2.1.2. Rice husk ash and lime

Rice husks were obtained from a paddy field in Quang Binh, Vietnam. Three different burning conditions were applied to produce uncontrolledly burnt ash, well-burnt ash and over-burnt ash. The incinerator used to burn the ash under controlled conditions had limited size and heating capacity. Accordingly, RHA is classified into three types:

- **U-RHA:** This ash was produced in uncontrolled conditions, by pouring the husks in an open fire. The ash was taken out of the fire when raw yellow ice husk was not seen anymore. Duration was less than 30 minutes.

- **500-RHA:** This ash was produced in optimized burning conditions suggested by Muthadhi and Kothandaraman (2010), which is a burning duration of 2 hours at 500°C. The incinerator was pre-heated to 250°C before putting a ceramic cup filled with rice husks in the incinerator. Due to the limited size of the laboratory incinerator the husks had to be burned in small batches of about 40 g to prevent oxygen limitations and allow for complete burning. It took about 30 minutes for the temperature to reach 500°C. The mass was then left for 2 hours and was quickly cooled down by immediately removing the cup from the incinerator. After about 15 minutes, the ash was transferred to a closed box and stored at room temperature.

- **700-RHA:** Rice husks were put in the incinerator at room temperature in batches of 100 g using a ceramic cup. Then the incinerator was turned on and set at 700°C. Without pre-heating it took about 2 to 3 hours to heat up to 500°C and about 5 hours to reach at least 650°C. The incinerator was turned off after 7 hours and the rice husk ash was kept in the incinerator overnight for cooling down. After burning the ashes were weighed

to calculate the ash content over the original RH, then it was ground using a vibration grinding mill (HSM 100, Herzog, USA) with a capacity of 10 ml, for 30 seconds to obtain fine ash. Dry powdered calcium hydroxide (lime) (Sigma-Aldrich, ACS grade, ≥96%) was used in both ash reactivity analysis and the soil improvement experiments.

2.1.3. Mixtures of soil, rice husk ash and lime

RHA was mixed with lime as a dry powder using a 1:1 mixing ratio. Then the mixture was added to the wet clay in varying amounts up to 6% of the wet clay weight. As a reference, also mixtures were prepared with only lime and only U-RHA at similar weight %.

2.2. Methods

2.2.1. Rice husk ash characterization

Several physical properties related to RHA reactivity were investigated:

(i) the surface area was determined according to the Brunauer–Emmett–Teller (BET) method using Gemini VII 2390, MicromeriticsR. Before measuring the specific surface area, samples were dried at 105°C for 2 hours;

(ii) crystal structure and mineralogical composition were analysed by X-ray diffraction analysis on a Bruker-AXS D5005;

(iii) particles were observed using an Environmental Scanning Electron Microscope, Philips XL30 ESEM ;

(iv) particle density was defined using a pycnometer filled with ethanol;

(v) elemental composition was analysed by using an XRF Spectrometer, Epsilon 3XL Panalytical.

The pozzolanic activity of the rice husk ash was evaluated using electrical conductivity measurement as proposed by Luxán et al. (1989): 5 g of ash was added to 200 ml of a saturated lime solution at 40°C, which was constantly stirred. Electrical conductivity and pH of the mixed solution at 40°C were recorded at 10 seconds intervals using a benchtop meter (C3010, Consort, Belgium)

Reactive silica content was identified according to EN 197-1 (CEN 2011), which is determined by subtracting the insoluble fraction of the ash from the total silica content. The total silica content was obtained from XRF results, and the insoluble fraction was determined according to section 10.2 of the standard EN 196-2 (CEN 2005), by treatment with hydrochloric acid (HCl) and potassium hydroxide (KOH).

2.2.2. Soil improvement evaluation

The water content and Atterberg limits of the various clay mixtures were determined according to CEN ISO/

TS 17892-12 (CEN 2004). Undrained shear strength was determined for various curing periods using the fall cone test according to ISO/TS 17892-6 (CEN 2004). After preparation the clayey mixtures were transferred in 100 mL plastic jars, which were covered by plastic foil and closed with plastic caps to prevent evaporation and stored at room temperature of 25°C. One-dimensional consolidation tests were performed to determine the compressibility of the clayey mixtures using a standard beam oedometer according to ISO/TS 17892-5 (CEN 2004). After the clayey mixtures were installed in the oedometer set up, the samples were left to cure for 7 days under a static load of 35kPa. After that, additional load steps were applied after the end of the primary consolidation phase was observed. In each step the previous load was approximately doubled.

3. Results

3.1. The properties of rice husk ash

After burning and cooling, the three ashes differed in colour: 500-RHA was grey, U-RHA was black, likely the result of unburned carbon, and 700-RHA appeared light pink (Figure 1). After grinding, the colour of 500-RHA and U-RHA remained unchanged, where 700-RHA turned grey.

The results of the other analyses are summarized in Table 1. XRD analysis (Figure 2a) showed that all the ashes



Fig. 1. Residual RHA after burning

Table 1
Determined rice husk ash properties

Type of rice husk ash		500-RHA	700-RHA	U-RHA
Crystal structure (XRD)		amorphous material and some quartz		
Ash content (Ash%)	[%]	12	12	21
Pozzolanic activity (PA)	[mS cm ⁻¹]	2.36	1.33	2.61
(EC reduction in first 2 min)				
Specific surface area (SSA)	[m ² g ⁻¹]	47	27	68
(Nitrogen Gas Adsorption)				
Particle density (PD)	[g cm ⁻³]	2.08	2.05	1.71
(pycnometer with ethanol)		(±0.09)	(±0.11)	(±0.05)
Silica content (Si%)	[% of total ash weight]	94.5	94.2	54.9
(XRF)		(±2)	(±2)	(±2)
Carbon content (C%) ^a	[% of total ash weight]	-	-	40.0
(XRF)				(±2)
Reactive silica content (Si ^R %)	[% of total ash weight]	93.6	92.8	52.1
(Boiling in KOH)		(±2.0)	(±2.9)	(±2.6)

^a derived from lost weight in XRF

were mainly amorphous, containing only a few small peaks which correspond to the mineral quartz. For all three types of ash, treatment with HCl and KOH resulted in dissolution of almost all the ash, indicating that most of the silica is reactive and therefore considered to be amorphous, which is consistent with the XRD analysis.

The ESEM image of a rice husk (Figure 3) showed that silica is concentrated on the outer epidermis of the rice husk, especially at protuberances and needle areas. In ESEM images of burned ashes, little differences were observed between 500-RHA and 700-RHA, except the large spherical particles of 700-RHA which seem to be products of sintering effects (Figure 2b and Fi-

gure 2d). In U-RHA, two different types of particles could be distinguished: dark and light grey (Figure 2c). As the greyscale in ESEM images is related to the material density, the dark particles are considered to be carbon where the light particles are silica.

All three types of ash showed good pozzolanic activity according to the classification provided by Luxán et al. (1989). As indicated in Figure 4, for all ashes the electrical conductivity reduced more than 1.2 mS/cm within the first 2 minutes after adding the ash to the lime solution.

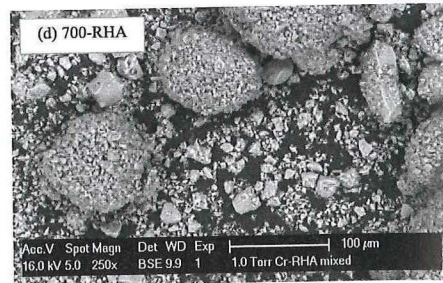
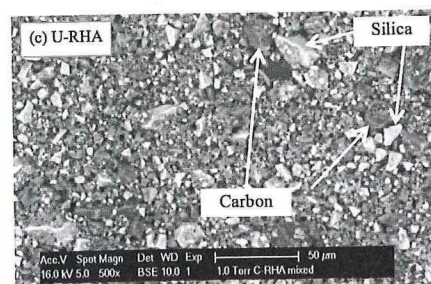
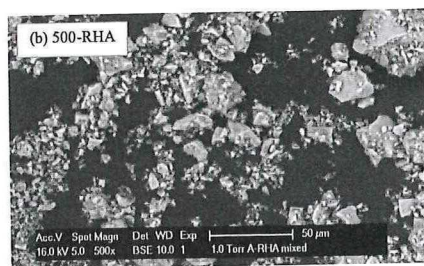
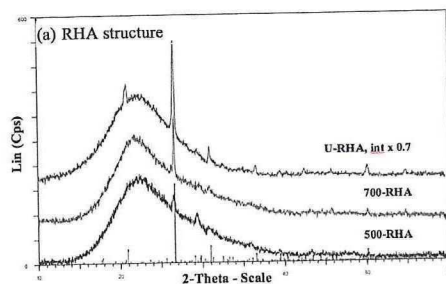


Fig. 2. RHA properties: the structure is mainly amorphous in all three types of ash; the particles are granular; sintering effect was seen in 700-RHA.

As expected, the 500-RHA, showed a higher reactivity than the 700-RHA. Unexpectedly, the U-RHA showed the highest reactivity. The silica content, according to XRF analysis, was about 94% for both 500-RHA and 700-RHA, and about 55% for U-RHA. The sum of all elements in the XRF

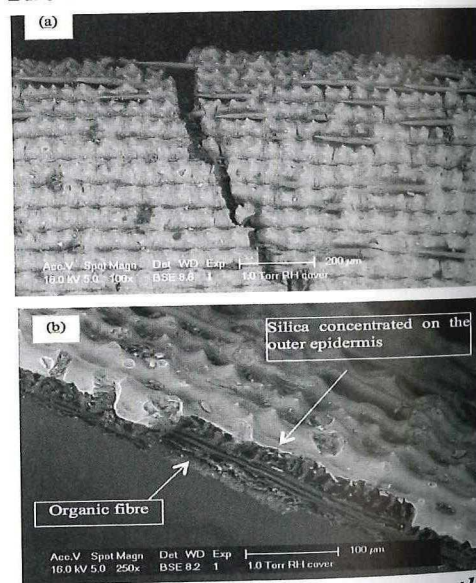


Fig. 3. Silica concentrates on the outer epidermis of a rice husk, observation at the outer surface (a) and at a cross section (b).

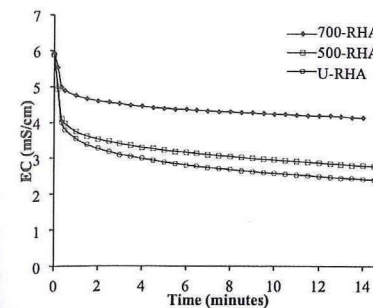


Fig. 4. Reactivity with saturated lime solution: all the RHA are reactive as their electrical conductivity reduction in the first 2 minutes is over 1.2 mS/cm; U-RHA is the most reactive.

for U-RHA did not reach 100% as it did for the other two samples, but only 60%. It was assumed that the missing other 40% in U-RHA was carbon, which could not be detected by the XRF methodology used in this study. Likely as a result of the considerable amount of carbon, U-RHA had the smallest particle density.

3.2. Results of soil treatment

3.2.1. Plasticity modification

Mixing the clay with lime and RHA immediately changed the consistency of the clay from soft to firm and lumpy. The significant increase in soil consistency is represented by a considerable reduction of liquidity index from 0.76 to nearly zero. The addition of dry additives decreased the water content of the soil from 48% down to about 43% depending on the amount of added material. However, the reduction in moisture content alone could not explain the change in soil consistency.

Figure 5a shows that both the liquid limit and the plastic limit were significantly affected by the addition of lime

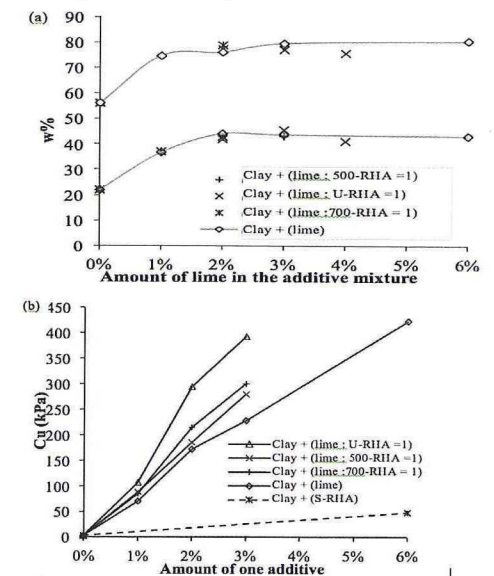


Fig. 5. Immediate and long-term influence of RHA on lime treatment for wet clay: (a) Similar immediate effect on the Atterberg limits of the treated clay, resulting in an improvement of its plasticity; (b) different effect of RHA on the development of undrained shear strength after 28 days; in combination with lime, U-RHA resulted in the largest improvement.

and RHA to the wet clay. Adding 2 % of lime to the clay increased the liquid limit from 56% to 76% and the plastic limit from 22% to 44%. As both liquid limit and plastic limit increased to the same extent the plasticity index remained more or less constant about 33%. Adding more than 2% of lime did not affect the plasticity limits any further. The change of plasticity limits

was mainly attributed to the lime. Adding RHA did not seem to have any additional effect on the plasticity limits nor did the type of RHA.

3.2.2. Consolidation behavior

Mixing the clay with the additives significantly changed the consolidation behaviour of the clay as shown in

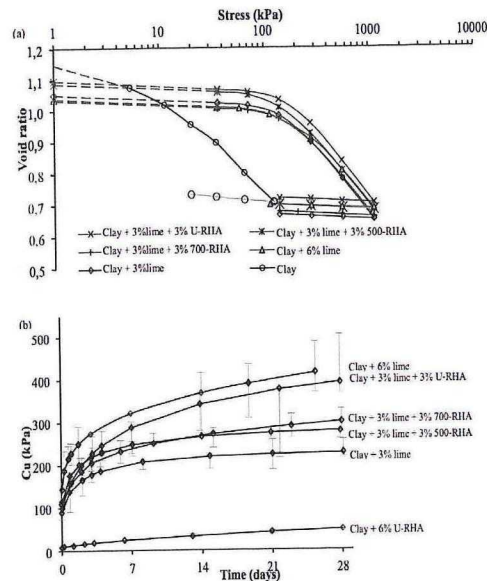


Fig. 6. Results when replacing 3% of lime by RHA: (a) similar improvement of consolidation characteristics of the clay; (b) different trend of strength development: U-RHA generated the most development.

Figure 6a. There was not much difference observed between the various type of additives nor between the various amounts of additives. In all the cases, the change in consistency after mixing and 7 days of curing caused the normal compression lines to shift to the right compared to the clay

without additives, resulting in an apparent pre-consolidation pressure of approximately 200 kPa. The compression index, C_c , was slightly affected by the additives, about 20% higher than for the clay without additives, while the unloading compression index, C_r , was reduced by a factor of two. The consolidation coefficient, c_v , for the clays with additives was a factor 20-30 higher than for the clay without additives (see Table 2).

3.2.3. Undrained shear strength

Although the addition of RHA to the lime treatment did not affect the results of soil consistency directly after mixing, it did show an effect on the undrained shear strength after curing. In all the cases with lime, the strength increased rapidly within the first day after mixing and kept increasing at least during the first week after which the strength increase gradually slowed down. When comparing the mixtures with 3% lime (Figure 6b), the sample with only lime showed an increase in undrained shear strength from about 100 kPa to about 200 kPa, while mixtures with lime and 500-RHA or 700-RHA increased from 100 to almost 300 kPa, and the lime and U-RHA showed the largest strength increase reaching 400 kPa, which was close to the strength increase of a sample with 6% lime without RHA. Mixtures with only RHA and without lime, did not show a significant strength improvement, as shown for a mixture with U-RHA.

Table 2

Effect of the different additives to the compressibility parameters

Soil	σ'_p (kPa)	C_c	C_r	σ' (kPa)	t_{50} (s)	C_v (m ² /year)	C_α $\times 10^{-3}$
Clay	17.1	0.344	0.033	35	2225	0.23	5.83
				64	1711	0.27	-
				124	1472	0.28	4.25
Clay + 3% lime	200.0	0.412	0.019	278	77	6.5	1.62
				557	68	6.6	2.10
				1104	67	5.9	2.16
Clay + 3% lime + 3% 500-RHA	172.2	0.419	0.020	277	98	5.1	1.49
				556	79	5.7	1.95
				1103	78	5.0	1.68
Clay + 3% lime + 3% U-RHA	215.8	0.441	0.018	276	73	7.0	1.99
				555	75	6.2	1.89
				1102	67	6.0	2.15
Clay + 3% lime + 3% 700-RHA	216.3	0.422	0.019	276	86	5.9	1.41
				550	68	6.8	1.95
				1097	61	6.6	2.16
Clay + 6% lime	232.5	0.407	0.014	279	77	6.7	1.26
				554	66	7.2	1.78
				1106	62	6.7	2.13

4. Discussion

4.1. The effect of burning conditions on rice husk ash reactivity

Overall, the properties of the rice husk ashes were in agreement with literature, except for the reactivity. The common color of completely burnt RHA is grey to white (Siddique 2008), which corresponds to the 500-RHA showing a light grey color after burning. The light pink color of 700-RHA was also reported by Rao et al. (1989) and Behak and Núñez (2013) as a result of excessive burning. The aggregation of particles which was observed in the ESEM images of ground 700-RHA could be attributed to the effect of sin

tering of the minor elements (Rao et al. 1989). The black colour of U-RHA is likely from black carbon, a typical product of incomplete combustion of organic materials, which occurs either when the burning duration is short or when the oxygen supply is limited. The lower density of black carbon compared to that of silica, might explain the lower particle density of U-RHA. Black carbon is also a highly porous compound with a large specific surface area (Donnet 1993), which could explain the large surface specific area of the U-RHA.

Comparing the reactivity of the rice husk ashes, the results were different

than expected. As the pozzolanic activity of rice husk ash is commonly attributed to the presence of reactive silica (Malhotra and Mehta 1996), it was expected that 500-RHA would be more reactive than 700-RHA, as higher burning temperature and duration would have caused the amorphous silica to crystallize. The pozzolanic activity measurement confirmed this expectation. However, both 500-RHA and 700-RHA showed a similar amount of reactive silica. Apparently the crystalline silica was not obtained in the process of 700-RHA. This might be because the temperature 700°C was not enough to form the crystalline silica. For example Behak and Núñez (2013) showed that at least 900°C was required to observe a decrease in reactivity. Although the reactive silica content was the same, the specific surface area for 500-RHA was much larger than for 700-RHA, which could explain the difference in reactivity. Dividing the

pozzolanic activity by the specific surface area showed that both 500-RHA and 700-RHA had similar reactivity to surface area ratios (Table 3), insinuating the higher reactivity of 500-RHA was the result of a larger specific surface area and not of a difference in reactive silica content. It could be that sintering of the particles at higher temperature and longer burning duration affected the grind-ability of the ash, resulting in the lower specific surface area, as observed in the ESEM analysis of 700-RHA.

The high reactivity of U-RHA was unexpected as the amount of reactive silica in the ash was much lower than in the other two ashes and the presence of carbon was considered to have adverse effect on the pozzolanic reactivity (Al-Khalaf and Yousif 1984), due to its interfering impact on the formation process of cementitious calciumhydrosilicates.

Table 3
Calculated rice husk ash properties

Type of rice husk ash		500-RHA	700-RHA	U-RHA
Pozzolanic activity per g of RHA (PA)	[mS cm ⁻¹ g ⁻¹]	0.47	0.27	0.52
Pozzolanic activity per m ² of RHA (PA/SSA)	[mS cm ⁻¹ m ⁻²]	0.0099	0.0100	0.0077
Pozzolanic activity per m ² of silica (PA/SSA/Si ^{R%}) ^{a,b}	[mS cm ⁻¹ m ⁻² -silica]	0.011	0.011	0.015
Pozzolanic activity per g of RH (PA/Ash%)	[mS cm ⁻¹ g ⁻¹]	0.056	0.032	0.109

^a assuming all pozzolanic activity in the RHA is attributed to silica and all other materials are inert.

^b assuming the fraction of surface area of each compound in RHA is proportional to its mass percentage.

A possible explanation for the high reactivity of U-RHA could be the large specific surface area. Similarly, Cordeiro et al. (2009) showed that high reactivity could be obtained for a RHA with a relatively high carbon content of about 12%, as long as the ash was sufficiently grinded to obtain a large specific surface area. Comparing the ratio between pozzolanic activity and the specific surface area, U-RHA showed a lower activity per square meter of ash than the other two types of ash (Table 3). This seems reasonable, considering that U-RHA has a significantly lower silica content and that the pozzolanic activity is attributed to the reactive silica.

However, assuming that 1) the pozzolanic activity is attributed only to the silica and all other materials are inert and 2) that the surface area of the silica, carbon and other elements is proportional to their mass percentage, the pozzolanic activity per square meter of silica can be calculated by dividing the pozzolanic activity by the specific surface area and the reactive silica content (Table 3). And based on these calculations the silica in U-RHA appears to be about 40% more reactive than in the other two ash types. The difference between the reactivity of the silica of U-RHA and the other two ashes could be even larger, considering the surface area of the silica and carbon is not necessarily proportional to their mass percentage. In fact, the relative surface area of silica can be considerably

smaller, due to the porous nature of the incompletely oxidized organic carbon, which can yield an extremely large specific surface area (Donnet 1993; Cordeiro et al. 2009). This result seems peculiar as the ratio between reactive and total silica did not significantly vary for the different ash types (Table 1). In fact, a lower reactivity of the silica was expected for U-RHA, considering that uncontrolled combustion causes a higher amount of crystalline silica due to locally high temperature values, as illustrated by the more pronounced quartz peaks in the XRD analysis (Figure 3a).

Another explanation for the high reactivity of U-RHA could be that the assumption that silica is the only reactive compound in the ash is not correct and that the carbon is contributing to the reactivity of the ash. It is well known that black carbon has good adsorptive properties (Donnet 1993), but so far no reports have been found attributing pozzolanic activity to black carbon. Overall it is clear that incomplete combustion by uncontrolled burning conditions led to a higher pozzolanic activity for U-RHA compared to the controlled burning conditions for 500-RHA and 700-RHA. On top of that, U-RHA has a higher yield, meaning more ash is produced per ton of rice husk: about 21% for U-RHA compared to about 12% for both 500-RHA and 700-RHA. As a result, the activity per gram of RH for U-RHA is almost twice as high as for 500-RHA and

more than three times as high as for 700-RHA (Table 3). Consequently, based on a higher pozzolanic activity and a higher product yield burning conditions leading to incomplete combustion and high carbon content seems to be the preferred method to produce highly reactive RHA.

4.2. The suitability of RHA for soil stabilization of clayey soils

The addition of lime and RHA to wet clay significantly affects the geotechnical properties. The results of experiments on treated soils showed that RHA needs lime to be activated, and when mixed with lime the ash contributed to enlarge the positive effect of the lime. The immediate effect of the additives - i.e. the increase in plastic and liquid limit and resulting increase in consistency, undrained shear strength and apparent pre-consolidation pressure - can be attributed exclusively to the addition of lime as directly after mixing, no significant differences were observed between the samples with lime only and mixtures of lime and RHA, nor between the different ash types. On the long term the addition of RHA to clay (in combination with lime) did have a significant effect on the development of undrained shear strength. Especially in the case of U-RHA, it can reduce the amount of lime required to reach a target strength significantly, e.g. for a strength of 400 kPa almost by a factor 2. The fact that lime is required to activate the siliceous RHA and that the effect of RHA on

strength development is only observed after significant curing time is similar to what is observed in concrete or cement and typical for pozzolanic materials (Hill et al. 1992).

Considering the effect of burning conditions on the suitability of RHA for ground improvement applications in clayey soils, the differences between the ash types in the geotechnical tests were not as large as expected from the activity measurement. No significant differences were observed between the three ash types in the geotechnical properties directly after mixing. After curing U-RHA showed a larger strength increase than the other two ash types, which seemed to correspond with the reactivity measurements. While the reactivity of U-RHA was slightly higher than 500-RHA, its ability to increase the strength seemed much better after curing than that of 500-RHA, despite the fact it had a much lower silica content. The difference in strength after curing between the other two ash types was negligible, even though 500-RHA showed significantly higher activity than 700-RHA. Based on the large strength increase after curing for U-RHA it seems strict burning conditions are not essential to produce highly reactive RHA and conditions of incomplete combustion might even be preferred when producing RHA for soil stabilization applications.

The clarification of these results and their implication for practical applica-

tions still requires further investigation. The results are somewhat contradictory with the general conviction that the carbon content of RHA should be minimized, as it would interfere with the chemical reaction producing the cementitious calciumhydrosilicates. Also it had been found that uncontrolled burning of rice husks in open fire or in industrial processes for rice parboiling or electricity generation results in RHA with relatively high carbon content and low activity. Behak and Núñez (2013) showed that residual RHA with high carbon content from a rice-parboiling plant was significantly less effective when mixed with lime as a stabilizing agent in sand compared with RHA which was prepared under controlled conditions and almost free of carbon. However, the presence of carbon is not necessarily the reason for the observed low activity of the residual ash as uncontrolled burning conditions can locally reach higher temperatures which promote crystallization of the silica reducing the pozzolanic activity. Also differences in specific surface area of the ash - which is significantly affected by the composition and the grinding procedure - and different mixing ratios of RHA and lime could be a reason for the observed differences in literature.

Applying uncontrolled burning to produce RHA for soil applications has both pros and cons that require evaluation at large scale. Its downsides are that it could lead to undesired gas

emissions such as carbon monoxide or volatile organic compounds, which are typical products of incomplete combustion and other byproducts of the oxidation process (Jenkins et al. 1998), while aiming for incomplete combustion might affect the efficiency of using rice husks as a fuel. On the other hand the addition of activated carbon to soils has additional positive effects besides the potential increase in strength and consistency as it improves fertility and forms a sink of carbon to mitigate the anthropogenic carbon dioxide emissions (Glaser et al. 2002).

5. Conclusions

The effect of burning conditions on the pozzolanic activity of rice husk ash (RHA) and its implication for stabilization of clayey soil was evaluated for three different burning procedures: uncontrolled burning in open fire and controlled burning at 500 and 700°C. Unexpectedly, uncontrolled burning produced RHA showed the highest activity and also the largest strength improvement when it was mixed with wet clay in combination with lime, despite a high carbon content. Based on a higher reactivity and larger product yield, incomplete burning appears more favourable to produce RHA for soil stabilization purposes, and strict burning conditions are not necessary.

Acknowledgements.

This work is sponsored by Agricultural Science and Technology Project VIE-2283 (SF), belonging to Ministry of Agriculture and Rural Development of Vietnam. It was executed at Delft University of Technology, in cooperation with Deltares. The authors sincerely thank the technicians of both the two institutes for their assistance to prepare the materials and implement the experiments. We also thank Prof. Ir. A.F. van Tol for his valuable comments and guidance.

References

- A. Seco, F. Ramirez, et al.** (2011). "Stabilization of expansive soils for use in construction." *Applied Clay Science* 51: 348-352.
- A.J. Choobbasti, H. Ghodrati, et al.** (2010). "Influence of using rice husk ash in soil stabilization method with lime." *Frontier of Earth Science, China* 4(4): 471-480.
- Agarwal, S. K.** (2006). "Pozzolanic activity of various siliceous materials." *Cement and Concrete Research* 36: 1735-1739.
- Al-Khalaf, M. N. and H. A. Yousif** (1984). "Use of rice husk ash in concrete." *International Journal of Cement Composites and Lightweight Concrete* 6(4): 241-248.
- Basha, E. A., R. Hashim, et al.** (2005). "Stabilization of residual soil with rice husk ash and cement." *Construction and Building Materials* 19: 448-453.
- Behak, L. and W. P. Núñez** (2013). "Effect of burning temperature on alkaline reactivity of rice husk ash with lime." *Road Materials and Pavement Design* 14(3): 570-585.
- CEN** (2004). Geotechnical investigation and testing. Laboratory testing of soil - Part 12: Atterberg limits. CEN ISO/TS 17892-12:2004.
- CEN** (2004). Geotechnical investigation and testing. Laboratory testing of soil - Part 6: Fall cone test. ISO/TS 17892-6:2004.
- CEN** (2004). Geotechnical investigation and testing. Laboratory testing of soil - Part 5: Incremental loading oedometer test. ISO/TS 17892-5:2004.
- CEN** (2005). Methods of testing cement. Part 2: Chemistry analysis, European Standard. EN 196-2:2005.
- CEN** (2011). Cement. Part 1: Composition, specification and conformity criteria for common cements, European Standard. EN-EN 197-1:2011.
- Cook, D. J.** (1986). *Rice husk ash. Cement Replacement Materials*. R. N. Swamy, London, Surrey University Press. 3.
- Cordeiro, G., R. Toledo Filho, et al.** (2009). "Use of ultrafine rice husk ash with high-carbon content as pozzolan in high performance concrete." *Materials and Structures* 42(7): 983-992.
- Cordeiro, G. C., R. D. T. Filho, et al.** (2011). "Influence of particle size and specific surface area on the pozzolanic activity of residual rice husk ash." *Cement and Concrete Composites* 33: 529-534.
- Donnet, J. B.** (1993). *Carbon Black: Science and Technology*, Second Edition, Taylor & Francis.

Corresponding author: Vinh Pham, M.Sc., PhD student
 p.v.pham-1@tudelft.nl
 T +31 (0) 624 178 966
 Geoscience & Engineering Department, Faculty of Civil Engineering and Geoscience, Delft University of Technology, Stevinweg 1
 2628CN Delft
 The Netherlands

- F. Haji Ali, A. Adnan, et al.** (1992). "Geotechnical properties of a chemically stabilized soil from Malaysia with rice husk ash as an additive." *Geotechnical and Geological Engineering* 10: 117-134.
- Glaser, B., J. Lehmann, et al.** (2002). "Ameliorating physical and chemical properties of highly weathered soils in the tropics with charcoal - a review." *Biology and Fertility of Soils* 35(4): 219-230.
- Handreck, K. A. and L. H. P. Jones** (1968). "Studies of silica in the oat plant - IV. Silica content of plant parts in relation to stage of growth, supply of silica, and transpiration." *Plant and Soil* 29(3): 449-459.
- Hill, N., S. Holmes, et al., Eds.** (1992). *Lime and Other Alternative Cements*. London, Intermediate Technology Publication.
- Jenkins, B. M., L. L. Baxter, et al.** (1998). "Combustion properties of biomass." *Fuel Processing Technology* 54(1-3): 17-46.
- Krishnarao, R. V. and M. M. Godkhindi** (1992). "Distribution of silica in rice husks and its effect on the formation of silicon carbide." *Ceramics International* 18(4): 243-249.
- Luxán, M. P., F. Madruga, et al.** (1989). "Rapid evaluation of pozzolanic activity of natural products by conductivity measurement." *Cement and Concrete Research* 19(63-68).
- Malhotra, V. M. and P. K. Mehta, Eds.** (1996). *Pozzolanic and Cementitious Materials. Advances in Concrete Technology*. Ontario, Canada, Gordon and Breach Publishers.
- Mehta, P. K.** (1979). *The Chemistry and Technology of cements made from rice-husk ash*. Proc. UNIDO/ESCAP/RCTT Workshop on Rice-Husk Ash Cement. Peshawar, Pakistan: 113-122.
- Muntohar, A. S.** (2002). "Utilization of Uncontrolled Burnt Rice Husk Ash in Soil Improvement." *Dimensi Teknik Sipil* 4(2): 100-105.
- Muntohar, A. S.** (2006). Swelling characteristics and improvement of expansive soil with rice husk ash. *Expansive soil: Recent advances in characterization and treatment*. Amer Ali Al-Rawas and M. F. A. Goosen, Taylor & Francis/Balkema: 435-452.
- Muntohar, A. S., A. Widiyanti, et al.** (2013). "Engineering properties of silty soil stabilized with lime and rice husk ash and reinforced with waste plastic fiber." *Journal of Materials in Civil Engineering* 25(9): 1260-1270.
- Muthadhi, A. and S. Kothandaraman** (2010). "Optimum production conditions for reactive rice husk ash." *Materials and Structures*(43): 1303-1315.
- Nair, D. G., A. Fraaij, et al.** (2008). "A structural investigation relating to the pozzolanic activity of rice husk ashes." *Cement and Concrete Research* 38: 861-869.
- Rao, G. R., A. R. K. Sastry, et al.** (1989). "Nature and reactivity of silica available in rice husk and its ashes." *Bulletin of Materials Science* 12(5): 469-479.
- Siddique, R.** (2008). *Rice Husk Ash. Waste Materials and By-Products in Concrete*, Springer Berlin Heidelberg: 235-264.
- UNIDO** (1984). *Rice Husk Ash Cements: their development and application*. Vienna.
- Van Tuan, N., G. Ye, et al.** (2011). "The study of using rice husk ash to produce ultra high performance concrete." *Construction and Building Materials* 25(4): 2030-2035.
- Walker, R. and S. Pavia** (2011). "Physical properties and reactivity of pozzolans, and their influence on the properties of lime-pozzolan pastes." *Materials and Structures* 44: 1139-1150.
- Yeoh, A. K., B. Rahim, et al.** (1979). The relationship between temperature and duration of burning of rice husk in the development of amorphous rice husk ash silica. Proc. UNIDO/ESCAP/RCTT Follow-up meeting on Rice husk ash cement. Alor Setar, Malaysia.