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Experimental study on influence of rice husk ash on mortar compressive strength at different temperatures

Nghiên cứu thực nghiệm ảnh hưởng của tro trấu tới cường độ của vữa ở các nhiệt độ khác nhau

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Abstract

An experimental study on compressive strength of mortar mixes containing different amounts of rice husk ash (5, 10, 20%) has been investigated. The impact of different elevated temperatures (150, 300, 450, 750°C) on mortar strength is also examined. The experiments found that RHA can be used in its untreated form up to 10% as cementitious replacement without detriment of mortar compressive strength. Furthermore, the addition of RHA improves the resistance of mortars at high temperatures excepting to the ones with replacement ratio of 20%.

Keywords: Compressive Strength; Elevated Temperature; Rice Husk Ash; Mortar;

Tóm tắt

Bài báo giới thiệu kết quả nghiên cứu thực nghiệm cường độ của vữa chứa ba hàm lượng tro trấu khác nhau gồm 5, 10, 20%. Ảnh hưởng của nhiệt độ tới cường độ vữa sau khi nung tại 150, 300, 450, 750°C cũng được nghiên cứu. Kết quả cho thấy có thể sử dụng tro trấu đốt tự do thay thế xi măng với hàm lượng 10% mà không làm giảm cường độ chịu nén của vữa. Ngoài ra, việc sử dụng tro trấu với hàm lượng nhỏ (5 và 10%) còn cải thiện khả năng chịu nhiệt của vữa.

Từ khóa: Cường độ chịu nén; Nhiệt độ cao; Tro trấu; Vữa;

1. Introduction

Concrete is recognized as one of the most widely used construction material. It is estimated that the average consumption of concrete is about 1 ton per year per every person on the planet [1]. However, the production of cement and concrete are associated with a significant environmental issue. Indeed, cement consists in concrete's ingredient that contributes most to its embodied energy. To produce 1 ton of cement in the optimal conditions, about 3 GJ of energy must be provided. In addition, cement is the largest source emission of CO_2, followed by aggregate, accounting for 74-81% and 13-20% of the total amount CO_2 yielded from the production of

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concrete [1]. Every kilogram of cement produced will release about 0.7-1.0 kg of CO_2 gas. The CO_2 emissions result from the calcite decomposition, the fuel combustion, and the transportation of cement between production and consumption sites, etc. Generally, the CO_2 amount released by the cement industry accounts for 5-7% of global emissions from all sectors [2].

According to Mehta [3], the demand for cement in the world is 1.6×10^9 t/year. Moreover, this demand is linearly increasing, especially in the developing countries like China, India, etc. Several solutions have been conducted to reduce the environmental impact of cement production, such as the change in the chemical composition of clinker, the use of alternative fuels, and other alternatives. One of the most promising solution is to minimize the clinker amount by maximizing the supplementary cementitious materials used in cement and promoting the use of blended cement [4]. According to Flower and Sanjayan [1], the CO_2 emission from concrete can be reduced by 22% in using 40% of blast furnace slag to replace cement and by 13-15% in employing 25% of fly ash to substitute cement.

Among the mineral admixtures produced annually, such as fly ash (500 million tons), limestone (170 million tons), blast-furnace slag (75 million tons), rice husk ash with an output of about 37 million tons represents a promising replacing cementitious material [5]. Along with wheat, rice is a popular crop in the world which plays an important role in providing food for population. Rice husk is a by-product of rice milling, accounting for about 20% of the 744.7 million tons of paddy produced annually in the world [6].

Rice husk ash (RHA) is solid residue derived from the burning of rice husk and accounts for about 25% wt. of the rice husk. The husk contains about 50% cellulose, 25-30% lignin, and 15-20% of silica [7]. When burning, cellulose and lignin will be burned and transformed into gas and leaving silica and some inorganic oxides. In order to generate the pozzolanic activity of rice husk ash, the silica must be amorphous.

The properties of rice husk ash strongly depend on the burning and grinding techniques (i.e. burning temperature, burning time, combustion environment, grinding method and duration). Several methods generating the ash from husk have been proposed, ranging from free-field combustion to fluidized-bed techniques [8, 9]. According to Mehta [10], amorphous silica would be formed primarily when the burning temperature was below 500°C in prolonged oxygen-rich conditions or when the combustion temperature is up to 680ºC but duration time is no longer than 1 minute. However, according to Yeoh et al. [11], rice husk ash can maintain its amorphous state at a burning temperature up to 900°C if the burning time is less than 1 hour, and the crystalline silica is formed at 1000ºC if the burning time is more than 5 minutes. Chopra et al. [12] observed that at burning temperature up to 700ºC, silica remains amorphous and crystalline silica will increase with time. James and Rao [13] recommended the lowest temperature to ensure complete combustion of organic matter is 402ºC.

The economic and social benefits of using rice husk ash as a cement replacement material have been recognized by various scholars. The RHA was used in concrete since 1924 with two German patents [14]. However, the use of RHA as a pozzolanic admixture has only really progressed since the early 70's [15]. Many works have reported that the addition of RHA permits the enhancement of the concrete properties including compressive strength and modulus of rupture [16-18], the reduction of concrete's permeability [16, 19], the increasing of chemical resistance [20], the reduction of the alkaline aggregate reaction ability [21] and the shrinkage minimization [17, 22].

Moreover; it is also shown that RHA can be used in high-performance and ultra-high performance concrete [5, 16, 18]. Nguyen et al. [5] concluded that it is able to make concrete with strength higher than 150 MPa using RHA with conventional method. This result gives an opportunity of replacing the silica fume. The silica fume is the traditional active admixture of UHPC which is expensive with a limited supply.

During the past decade, the research on mortar or concrete mixtures incorporating RHA has attracted the attention of various scholars. Rukzon and Chindaprasirt [23] studied the strength and carbonation resistance of mortar mixture that employs portland rice husk ash cement. As a result, this works demonstrated that the inclusion of rice husk ash produces mortar mixtures with good strength and low porosity. Antiohos et al. [24] examined the pozzolanic properties of untreated RHA and its impact on the mortar strength, capillarity absorption, permeability and diffusion.

On the basis of the above literature review, despite the fact that a lot of works have been done on treated RHA, studies on the direct uses of untreated RHA are still limited and are usually dedicated to strength contribution, durability aspects but not to the behavior at elevated temperature. In this work, the utilization of RHA as a supplementary cementing material in its untreated form is examined. Our study response to the industry's increasing awareness for a more eco-friendly cement-based material. RHA was examined with respect to its inherent characteristics and its impact on the performance of cement-based mortars. Strength and behavior at elevated temperatures were investigated. Taken into account that during their service life, building materials may be subjected to accidental fires, their resistance to extreme temperatures seems to be a parameter that should be further investigated. In fact, it's interested in knowing the resistance of building material exposed to high temperature in case of burning.

2. Research method

In this study, Portland cement with the strength grade of 50 complying with the Vietnamese standard TCVN 2682-2009 [25] was employed. CEN standard sand according to the requirements of EN 196-1 [26] was used as the fine aggregate. This was natural, siliceous sand consisting preferably of rounded particles and has a silica content of at least 98%. The moisture content is less than 0.2% wt. of the dried sample.

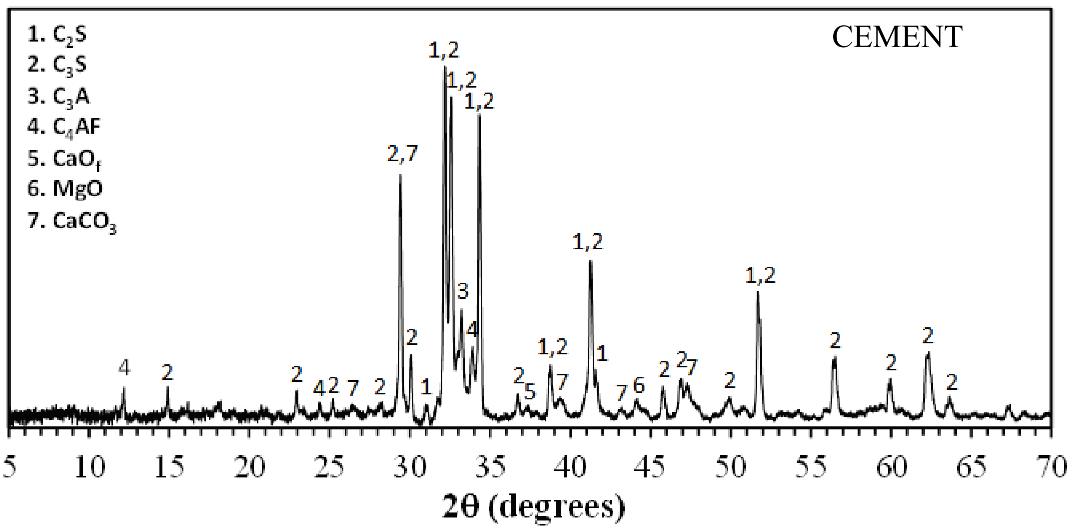
The rice husk ash (RHA) was obtained by free-field combustion of rice husk collected in Quang Nam province (Vietnam). Then RHAs was sieved to eliminate the grains with size larger than 0.09 mm. The chemical compositions of cement and rice husk ash were analysed according to TCVN 141:2008 [27] Vietnamese standard and summerised in **Table 1**. The loss of ignition (LOI) is equal to 3.97, implying that the amount of unburnt carbon existing in the ash is insignificant, thus resulting in light grey color, moderate water absorption.

Table 1. Chemical compositions and physical properties of cement and rice husk ash

Properties	Rice husk ash (%)	Cement (%)
CaO	4.23	59.06
SiO_2	87.30	21.14
Al_2O_3	0.54	4.79
Fe_2O_3	0.48	3.70
MgO	1.22	1.95
SO_3	-	2.14
LOI	3.97	3.68
Fineness (BET) (m^2/g)	22.17	2.11

In addition, XRD analysis was performed by means of Rigaku SmartLab X-ray diffractometer (Cu-Kβ radiation, 40 kV, 30 mA) and the obtained results are displayed in the **Fig.1**. It can be seen that the principal phases of cement are composed of calcium silicates and aluminates. For the RHA, the presence of crystalline silica with the peaks of quartz and cristobalite is recognized.

Mortars were composed of cement, sand and water. The cement was replaced by RHA at four different ratios by mass including 5%, 10%, 20% and 30% to investigate the influence of rice husk ash on the mortars properties. The sand to binder (cement + RHA) ratio was kept constant and equal to 3. The water to binder ratio was of 0.6. Because of high water absorption capacity of rice husk ash resulting from its porous nature, a polycarboxylate based superplasticizer was used to ensure a similar workability of all mortars. Workability target was set at a slump of 55 ± 5 mm measured by a mini cone [25]. Detail composition of studied mortars was given in the **Table 2**.



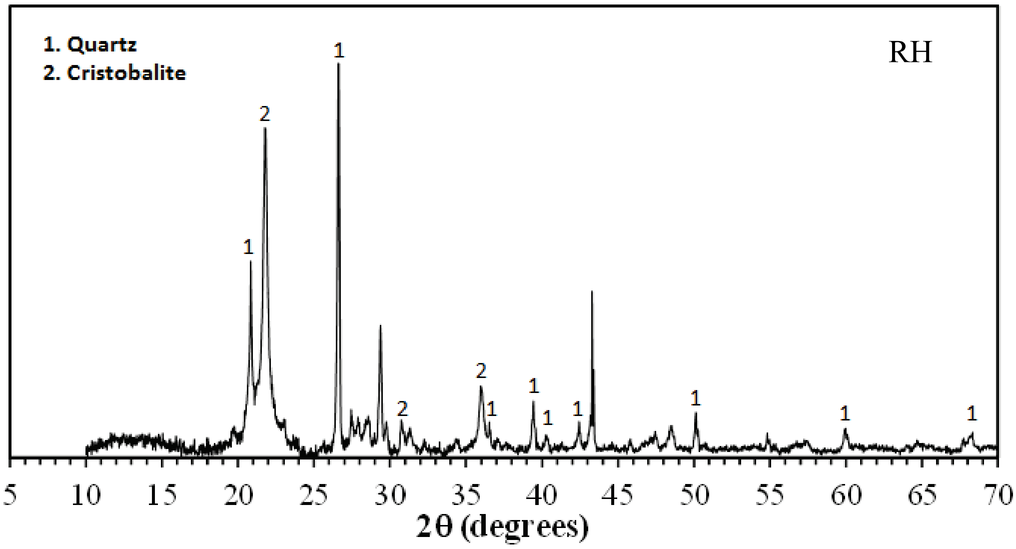


Fig 1. XRD patterns of cement and rice husk ash

Table 2. Mortar compositions

Sample	Cement (g)	RHA (g)	Sand (g)	Water (g)	Superplasticizer (mL)
0%	450	0	1350	270	0
5%	427.5	22.5			0.08
10%	405	45			0.32
20%	360	90			1.04
30%	315	135			2.40

The mortars were made in mixer with a capacity of 5 L, and using two different speeds (low and high). The mixing procedure used in all tests was as follows: pouring carefully dry mixture of cement and RHA in the mixer bowl which has contained already water (with the addition of superplasticizer in the case of using RHA). The paste was immediately mixed in 60 seconds at low speed (140 rpm). During the last 30 seconds, the sand was gradually added in the paste. Then, the mortar was mixed in 30 seconds at high speed (285 rpm) followed by a 90 seconds pause. And the mortar was mixed for 60 more seconds at high speed. After mixing, the samples were cast in the mould of 40×40×160 mm^3 and compacted by tamping the mortar 120 times in two layers.

The mortar samples were then cubed at 27±2°C and 100% relative humidity until compressive strength test (3, 7, 21, 28, 56 days) or residual compressive strength test after heating at elevated temperatures (56 days). The average compressive strength was determined according to the TCVN 3121-11:2003 [28] Vietnamese standard on the six specimens. These were obtained from three specimens of 40×40×160 mm^3 after the flexural strength test.

Mortar samples at 56 days age were calcined at 150°C, 300°C, 450°C, 750°C to evaluate the effect of RHA on the mortar fire resistance. These temperatures correspond to the ones at which the water evaporates, CSH gel, ettringite, monosulfate, porlandite decompose. The oven used is Nabertherm (LE6/11/R7) which can reach 1300°C au maximum. For each temperature grade, each mortar has tested on 6 samples.

Concerning the firing process, the oven is set up from the ambient temperature to the desired temperatures (150°C, 300°C, 450°C, 750°C) at a rate of 0.5°C/min, maintained at that temperature for 2 hours. Such an increasing of heating rate allows ensuring a uniform temperature in samples. After exposure to elevated temperatures, the samples were left in the furnace to cool gradually down to room temperature in order to avoid any temperature shock. Within 2 hours after achieving ambient temperature, the samples were brought out from the furnace and tested in compression to determine the residual compressive strength by means of an unstressed residual strength test according to the TCVN 3121-11:2003 Vietnamese standard.

Table 3. Mortar compositions

Variables	Unit	Min	Average	Std.	Max
Rice husk ash replacement percentage	%	0.00	13.00	11.00	30.00
Cement	g	315.00	391.50	48.47	450.00
Rice husk ash	g	0.00	58.50	48.47	135.00
Superplasticizer	mL	0.00	0.77	0.89	2.40
Heating Temperature	°C	27.00	198.33	242.01	750.00
Age	day	3.00	37.67	21.59	56.00
Compressive strength	MPa	0.00	42.99	22.24	75.02

3. Experimental result and comparison

Regarding the compressive strength of the RHA modified samples, the experimental results determined from six specimens corresponding to 3, 7, 21, 28 and 56 days testing time are shown in the **Fig. 3**. It can be seen that all the mortars strengthen with the hydration time. The compressive strength of mortars prepared with 5 and 10% RHA as partial replacement of cement evolves similarly as the mortars without RHA. Between 7 and 21 days, the strength growth rate is improved in the slowest manner. After 56 days, the mortars with 5% and 10% RHA reaches about 53 MPa which equals to about 93% of the control mortars.

When the RHA content is increased up to 20 and 30%, the mortars behavior differs totally. During the first 28 days, the strength of these two mortars evolves moderately, reaches only about 30% of the final strength at 56 days. However, their compressive strength is enhanced remarkably in the next 28 days. The 20% RHA contained-mortars achieve almost the same compressive strength by compared with the two other mortars containing 5% and 10% RHA. The resistance to compression of the 30% RHA contained-specimens reaches 82% in comparing with the control specimens.

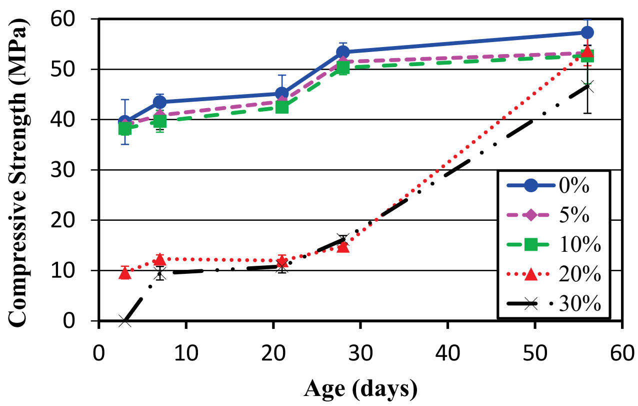


Fig. 2. Compressive strength of mortars at 27°C

To provide a better comparison, the normalized strength results relative to the control mix are presented in **Fig 3**. It can be inferred from the results that the addition of RHA does not imply any effect of strength improvement. These observations are coherent with those obtained by previous studies [24, 26]. In fact, the rice husk ash used in this study is in untreated form which provides a low amorphous silica quantity and specific surface. The filler effect of mineral admixture for a more compact package is not witnessed, the hydration of alite is so not enhanced and the pozzolanic activity of rice husk ash is moderate. However, there is not clear explanation on the very satisfactory early-age performance of RHAs in specimens 5 and 10%, given that their untreated nature cannot provide an adequate filler effect and that reactive silica cannot provide significant strength contribution unless hydration is at a progressed state [24, 27].

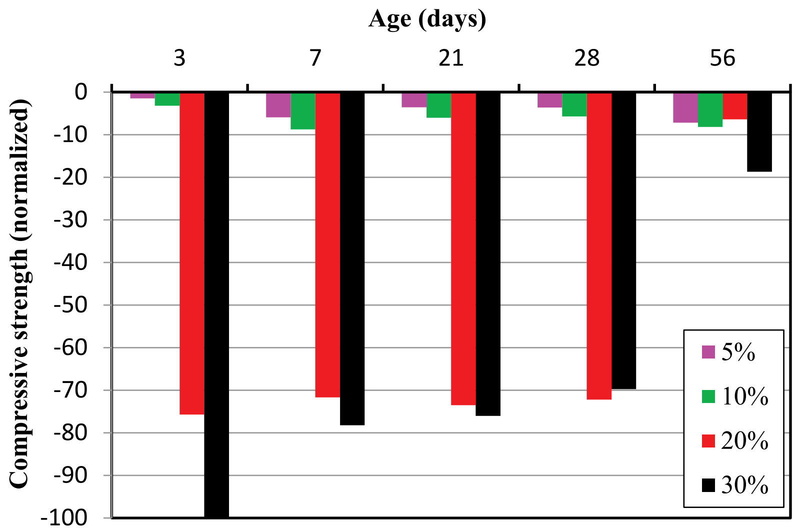


Fig. 3 Normalized compressive strength results relative to control mix

The effect of rice husk ash on the behavior of mortar specimens after exposed to different temperatures (27, 150, 300, 450, 750ºC) is investigated. Visually, it can be seen that the mortar samples are whitened after heating. At the temperature of 450ºC, some minor peelings are observed on the sample surfaces. When mortar samples heated to 750ºC, they appear crazing throughout the surface.

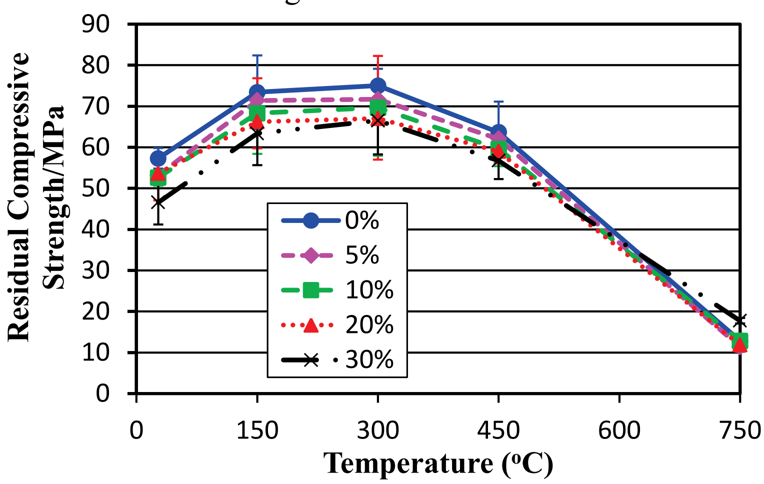


Fig. 4 Residual compressive strength of mortars versus thermal loads

Fig. 4 represents the residual resistance to compression of mortars subjected to elevated temperatures. It can be seen that after exposing to elevated temperature up to 450ºC, the mortars were strengthened. This may be due to the hydration of unhydrated cement particles. The water (in the form of steam) is eliminated intensively, and affects the surrounding phases of cement paste. Mainly due to flow resistance and high temperature, water vapor creates a high pressure in the paste. In consequence, the so-called condition for internal autoclaving appears in the cement paste and the result is an additional hydration of unhydrated cement grains. For the pozzolanic material, an additional tobermorite gel is formed as a result of the pozzolanic reaction of $Ca(OH)_2$ in OPC, with reactive silica in pozzolanic material [28]. Rashad et *al.* also indicates a residual compressive strength increase of paste containing ground granulated blast-furnace slag for temperatures up to 600ºC [29].

With further increasing of temperature up to 750ºC, the mortars show severe deterioration in residual compressive strength. In fact, the bond between the neat cement paste and the aggregate is weakened because the paste contracts following water loss, while the aggregate expands. In addition, at approximately 573ºC, the allotropic transformation of quartz-α into quartz-β occurs with an expansion. Furthermore, the decomposition of CSH compound tends to a loss of cementing ability in mortar. It was explained that the decomposition of CH into CaO and its subsequent reaction with the humidity from the surrounding air during cooling process cause micro-cracks resulting from the volume increase associated with this process.

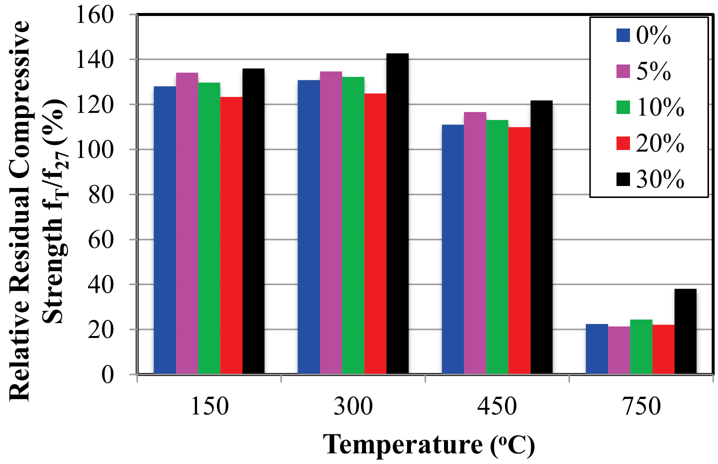


Fig. 5 Relative residual compressive strength of mortars versus thermal loads

The residual compressive strength after heating at different temperatures T is expressed as a ratio f_T/f_{27}, where f_T is the strength after heating at TºC and f_{27} is the initial strength of concrete at 27ºC. The strength ratio f_T/f_{27} as a function of the specimens' temperature T is shown in **Fig. 5**. The extent of improvement in compressive strength is reduced with the increase in the RHA content except the 30% RHA samples. The samples with 20% RHA show the worse capacity to high temperature than the control ones. This addition rate is sensitive. In reverse, the fire resistance of the highest modified RHA samples is enhanced the best.

4. Conclusion

Motivated by the economic and environmental benefits of using RHA as a cement replacement material, this paper has carried out an experimental work to investigate the durability, reflected by compressive strength, of mortar mixes containing RHA. The experiments found that RHA can be used in its untreated form up to 10% as cementitious replacement without detriment of mortar compressive strength. Furthermore, the addition of RHA improves the resistance of mortars at high temperatures excepting to the ones with replacement ratio of 20%.

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