Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2016, 8(2):114-122

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Effects of rice husk ash as filler on the bond strength and mechanical properties of ceramic tile mortar

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ABSTRACT

This work investigated the applicability of rice husk ash as filler in ceramic tile mortar. Rice husks, obtained from a rice mill in Abakaliki were washed, dried and heated to char (carbonized) on a gas stove until there was no emission of fumes. The rice husk char obtained was incinerated under controlled conditions in a muffle furnace at 650°C for four (4) hours to produce rice husk ash (RHA). The ash obtained was ground to reduce the particle size and then sieved with a standard sieve to obtain particle size of ~187µm which was used as filler in ceramic tile mortar. The mechanical properties of the prepared mortar were determined and compared with those produced with $CaCO_3$ as well as Top Fix, a popular brand in the Nigerian market, used as reference standard. The results showed that the RHA-filled mortar had higher bond strength (11.65 KPa), compressive strength (1290 KPa), and tensile strength (165 KPa) than the samples formulated with $CaCO_3$. However RHA-filled mortar showed lower flexural strength than the $CaCO_3$-filled samples. Rice husk ash mortar was found to be comparable to Top Fix, the commercial product.

Key words: Agro waste, Rice husk ash, Filler, Mortar, Ceramic tile, Bond strength

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INTRODUCTION

Mortar is a workable paste used to bind building blocks such as stones, bricks, and concrete masonry units together, fill and seal the irregular gaps between them, and sometimes add decorative colours or patterns in masonry walls. In its broadest sense mortar includes pitch, asphalt, and soft mud, cement paste or clay, such as used between bricks [1]. Ceramic tile mortar refers to the cementitious admixture used as adhesive in laying ceramic tiles. Ceramic tiling is commonly used these days in various application fields inside and outside buildings especially on floor and wall covering of building and swimming pools. The major materials involved in tiling are ceramic tiles, adhesive mortar [2] and the substrate (floor or wall). In many modern construction applications, pure cement-based mortars without organic polymer binders are not able to meet the state-of-the-art technical requirements [3]. Even mortars that contain cellulose ether additives to improve water retention capability and thus curing of the cement, adhere poorly, or not at all, on many of the materials used in the modern construction industries like polystyrene, cement, fibre and wood panels; as well as non-absorbent substrates like old tile surfaces and fully vitrified tiles [3]. Furthermore, cement-based mortars are very hard, brittle and nonflexible materials; and in many applications, flexible and deformable cement mortars are needed. Thus, the modification of cement-based mortars with polymers and other additives is, today, essential for many mortar applications [3].

Generally, the main dry components of a ceramic tile mortar are cement (inorganic binder), mineral fillers, fine aggregates and organic additives, mainly cellulose ether and redispersible polymer powder (RPP). Cellulose ethers are part of the mortar formulation mostly due to workability reasons as they improve water retention, act as thickening agent and introduce air voids into the fresh mortar [4][5][6]. They as well influence cement hydration, exhibiting a retarding effect on the drying of mortars [7]. Aside water, a typical formulation for ceramic tile adhesive according to Jenni et al [8] comprises ordinary Portland cement (35%), 425 - 500μm-size quartz sand (40%), calcium carbonate powder (22.5%), cellulose ether (0.5%) and redispersible polymer powder (2%) [8].

Industrial formulations are usually more complex and contain additional components [9]. In a so-called two-binder system, the mineral binder (e.g. cement) and the polymer binder complement each other ideally. Their combination results in outstanding synergistic properties and characteristics of the dry mortar, which cannot be produced by either of the two individual binders alone. Optimizing the formulation or enhancing the system components continuously improves adhesive mortar properties. One of the most important relatively non-adhesive additives in ceramic tile mortar formulation is the filler. Fillers are generally added in ceramic tile adhesive formulations to provide some of these simultaneous benefits [3]: enhance the interactions between the inorganic binder and polymer binder by way of modifying the flow properties of the adhesive, degree of cement hydration, the setting time and bond strength of the admixture; creates reinforcement between the adhesive and the adherends, increases resistance to environmental degradation, reduces shrinkage and stress during cure, extends pot life and reduces cost [3][5]. Substances often used as fillers in ceramic tile mortar are silica sand, limestone sand, dolomite sand, marble sand [10], silica fume[11], fly ash[12], composite clay and calcium carbonate[8].

Researchers, in their effort to convert wastes to useful materials, have since many past decades been investigating the use of agro wastes (plant and animal by-products) as filler materials in cement, concrete, thermoplastic thermosetting and other adhesive composites. Several reports have been made on the improvement of the rheology[13], the adhesion and mechanical properties of adhesives via the incorporation of agro wastes as filler materials [14][15][16][17][18]. Rice husk is generated in large quantities in the rice producing areas of the world during the process of rice milling. The domestic and industrial demand for rice husk is extremely low, thus large volumes of unutilized rice husk in these areas constitute an environmental nuisance [19]. Therefore the use of rice husk ash for industrial application will go a long way in minimizing the accumulation of rice husk in the environment.

Filler applications of rice husk ash (RHA) in plastics, elastomers and other polymer composites have been reported. RHA has been reported as excellent filler in natural rubber, epoxidized natural rubber [20][21][22], polypropylene/ethylene-propylene-diene terpolymer [23][24][]25], and epoxy foams [26]. RHA has been reported as adsorbent in water treatment and vegetable oil treatment [27]. As a pozzolan, RHA has been used in the glass, ceramics and cement industries [28]. The basic uses of RHA in the cement industry include: manufacturing of low cost building blocks and production of high quality cement [22][29]. The addition of RHA to cement has been reported to speed up setting time, improve compressive strength, improve resistance to acid attack and improve resistance to chlorine penetration in case of use in marine environment [30][31][32][33][34]. Recently, it has been used as extender in red oxide primer [19] and textured paint [36], in cellulose matt paint [37] and acrylics/poly vinyl acetate/natural rubber wood adhesives [38].

In this work, the effects of RHA in the form of amorphous silica [37] as filler on the bond strength, compressive strength, tensile strength and flexural strength of ceramic wood adhesives were investigated and compared with adhesive mortar formulated using $CaCO_3$ as filler.

EXPERIMENTAL SECTION

2.1 Materials

Cement: BURHAM cement was purchased from Ebonyi State Building Materials Market, Abakalik, Ebonyi state, Nigeria. It is an ordinary Portland cement that conforms to Nigerian standard with a Blaine specific surface area of 467.9m^2/kg, setting time of 75minutes, a specific gravity of 3.12 and compressive strength of 27.5, 42 and 58 (Mpa) at 2, 7 and 28 days, respectively (according to the producer).

Quartz sand: Natural quartz sand was obtained from Akpoha River in Afikpo North Local Government Area of Ebonyi State in Nigeria. The quartz sand used has 0.5mm maximum diameter and is classified as very fine sand [11].

Acacia gum and Carboxy methyl cellulose (CMC): These were supplied by Glisco Laboratory Chemicals, Enugu state, Nigeria. Acacia gum has been reported as a good substitute to petroleum-based RPP [39], and it was applied thus.

2.2 Equipment
i. Compressor (Wallace C862030)
ii. Tensometer (IT15MAT20)
iii. Flexometer (Wallace C82075)

2.3 Compounding of Ceramic Tile Mortar
The formulations used in this study are modification of the method proposed by Jenni et al [8]. Calcium carbonate is the standard filler used in the production of ceramic tile adhesive mortar. In this study, however the use of RHA as a substitute was investigated. A total of sixteen (16) samples of ceramic tile mortar (CTM0C - CTM16C and CTM0R - CTM16R) were produced with variation in the types of filler ($CaCO_3$ or RHA) and levels of filler. Table 1 and 2 show the ceramic tile mortar formulated with variation of filler levels of $CaCO_3$ and RHA respectively. The filler level was at the expense of cement ratio. The ratios of other dry components: quartz sand, cellulose ether and acacia gum were kept constant. The quality parameters of the RHA-filled adhesives were compared with those of $CaCO_3$-filled and with that of Top Fix, a popular commercial brand in Nigeria.

Table 1: Formulation of ceramic tile mortar (dry mix) using varied wt% of $CaCO_3$ filler

Component	CTM0C	CTM4C	CTM6C	CTM8C	CTM10C	CTM12C	CTM14C	CTM16C
Cement	47.0	43.0	41.0	39.0	37.0	35.0	33.0	31.0
Quartz sand	52.0	52.0	52.0	52.0	52.0	52.0	52.0	52.0
$CaCO_3$	0.0	4.0	6.0	8.0	10.0	12.0	14.0	16.0
CMC	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Acacia gum	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Table 2: Formulation of ceramic tile mortar (dry mix) using varied wt% of RHA filler

Component	CTM0R	CTM4R	CTM6R	CTM8R	CTM10R	CTM12R	CTM14R	CTM16R
Cement	47.0	43.0	41.0	39.0	37.0	35.0	33.0	31.0
Quartz sand	52.0	52.0	52.0	52.0	52.0	52.0	52.0	52.0
RHA	0.0	4.0	6.0	8.0	10.0	12.0	14.0	16.0
CMC	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Acacia gum	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

CTM4C = ceramic tile mortar with 4% of $CaCO_3$
CTM4R = ceramic tile mortar with 4% of RHA

The quality parameters tested on the formulated ceramic tile mortar variants are bond strength, compressive strength, tensile strength and flexural strength. The values of these parameters determined were compared with those of Top Fix.

2.4 Testing of Ceramic Tile Mortar Quality Parameters
i. Bond Strength
Standard test methods such as BS5980:1980 provides general evaluation of tile adhesives bond strength under laboratory conditions [38]. However, pull-off test which can be classified as a near- to-surface, partially destructive method was improvised to evaluate the adhesion of the tiling system. In this method, bond strength was determined based on modified BS 5980:1980 principles. Concrete slabs with dimensions of 350mm x 200mmx70mm were prepared. Ceramic tiles of 350mm x 200mm size were cut. The tile mortar was thinly spread on a section (200mm x 200mm) of the slab surface leaving part of the surface (150mm x 200mm) free. The tile was laid on the concrete surface on which the mortar had been applied.

The free end of the slab was clamped against a table top and force (load) was applied via a loop around the free end of the tile. The force was gradually increased until at a point when pull-off between the two surfaces was noticed. The value of the force, x, was regarded as the mortar bond strength expressed in ($\frac{N}{40000mm^2}$).

The value of x was converted to Y expressed in paschal ($\frac{N}{m^2}$) using the following formula:

Y =25x
Where x = $\frac{N}{40000mm^2}$ (measured)
Y = $\frac{N}{m^2}$ (S.I unit of pressure)
25 = constant (factor).

ii. Mechanical Properties
Maintaining constant water content of 25.5 ml to 100g of dry mix, a set of 16 samples of ceramic tile mortar mixtures was prepared. The compressive strength of the adhesive mortar was determined with Wallace C862030 Compressor, the tensile strength was measured using IT15MAT20 Tensometer and the flexural strength was obtained using Wallace C82075 Flexometer. The tests were conducted after 2 days, 7 days and 28 days of application of the mortar as shown in Tables 3 - 6 and Figs.1- 4.

RESULTS AND DISCUSSION

Table 3: Bond strength (KPa) of $CaCO_3$-filled, RHA-filled and TF ceramic tile mortar

% filler	2 days		7 days		28 days	
	$CaCO_3$	RHA	$CaCO_3$	RHA	$CaCO_3$	RHA
0	6.70 ±0.03	6.70 ±0.02	9.10 ±0.01	9.10 ±0.04	11.40 ±0.03	11.40 ±0.01
4	6.25 ±0.01	6.25 ±0.06	9.05 ±0.10	9.13 ±0.01	11.40 ±0.02	11.45 ±0.01
6	6.30 ±0.01	6.35 ±0.01	9.05 ±0.07	9.13 ±0.01	11.43 ±0.01	11.48 ±0.04
8	6.40 ±0.01	6.44 ±0.01	9.13 ±0.06	9.20 ±0.03	11.48 ±0.05	11.48 ±0.01
10	6.40 ±0.05	6.45 ±0.03	9.03 ±0.01	9.25 ±0.03	11.45 ±0.01	11.65 ±0.03
12	6.30 ±0.03	6.45 ±0.01	8.98 ±0.02	9.25 ±0.01	11.45 ±0.01	11.65 ±0.01
14	6.10 ±0.01	6.38 ±0.04	8.80 ±0.05	9.18 ±0.06	11.13 ±0.02	11.45 ±0.01
16	6.00 ±0.02	6.30 ±0.01	8.68 ±0.03	9.13 ±0.02	11.05 ±0.01	11.40 ±0.03
TF	6.50 ±0.03	6.50 ±0.05	9.20 ±0.01	9.20 ±0.01	11.60 ±0.03	11.60 ±0.05

Table 4: Compressive Strength (KPa) of $CaCO_3$- filled, RHA-filled and TF ceramic tile mortar

% filler	2 days		7 days		28 days	
	$CaCO_3$	RHA	$CaCO_3$	RHA	$CaCO_3$	RHA
0	560	560	1120	1120	1200	1200
4	510	560	1060	1130	1200	1240
6	510	570	1060	1130	1190	1260
8	530	580	1060	1160	1190	1260
10	540	590	1100	1180	1200	1290
12	550	590	1090	1170	1540	1290
14	510	570	1060	1140	1212	1270
16	488	550	1000	1110	1204	1250
TF	600	600	1180	1180	1280	1280

Table 5: Tensile strength (KPa) of $CaCO_3$-filled, RHA-filled and TF ceramic tile mortar

% filler	2 days		7 days		28 days	
	$CaCO_3$	RHA	$CaCO_3$	RHA	$CaCO_3$	RHA
0	156	156	160	160	162	162
4	154	152	156	158	159	160
6	154	154	156	158	159	160
8	155	154	156	159	160	162
10	157	156	158	160	160	165
12	156	156	158	162	160	163
14	154	153	156	157	160	162
16	154	153	155	156	157	161
TF	157	157	160	160	164	164

Table 6: Flexural strength (KPa) of $CaCO_3$-filled, RHA-filled and TF ceramic tile mortar

% filler	2 days		7 days		28 days	
	$CaCO_3$	**RHA**	**$CaCO_3$**	**RHA**	**$CaCO_3$**	**RHA**
0	210	210	240	240	290	290
4	210	200	240	230	290	260
6	220	200	250	230	290	250
8	220	190	250	200	300	250
10	230	180	250	190	300	240
12	220	170	250	190	300	230
14	192	164	230	170	280	230
16	180	156	220	160	250	200
TF	220	220	240	240	280	280

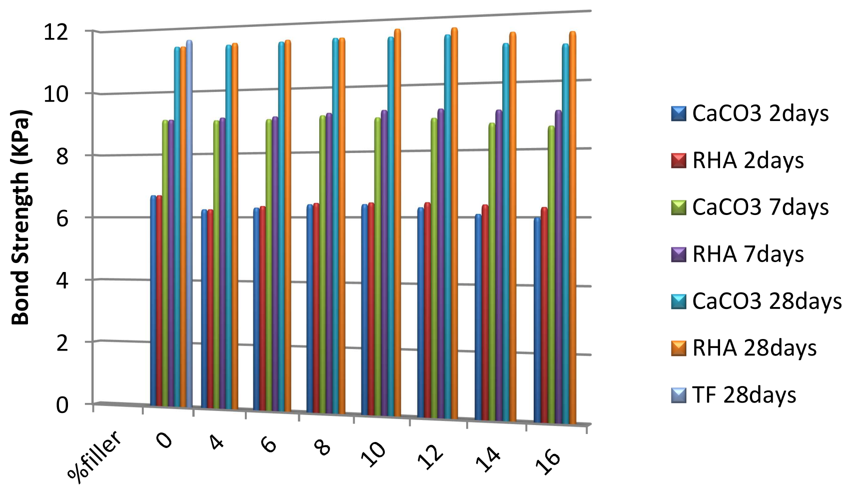

Fig.1: Plot of bond strength versus weight% of $CaCO_3$ and RHA fillers in CTM

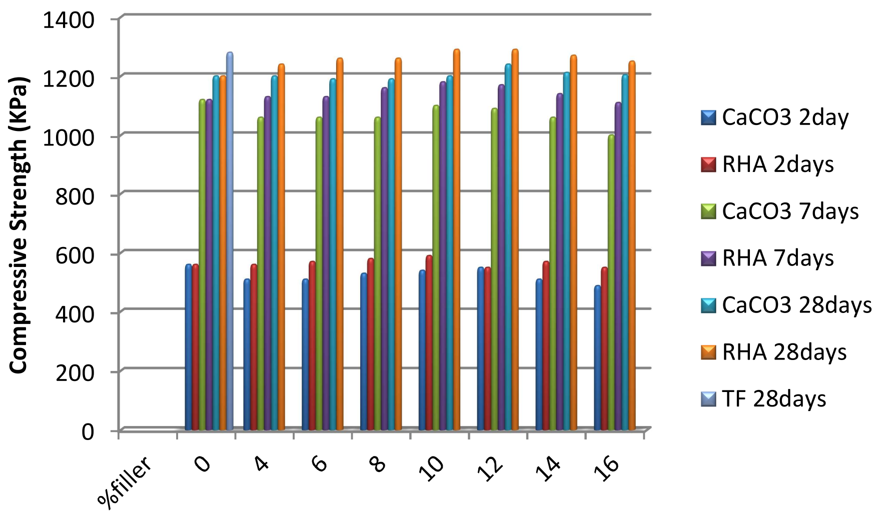

Fig.2: Plot of compressive strength versus weight% of $CaCO_3$ and RHA fillers in CTM

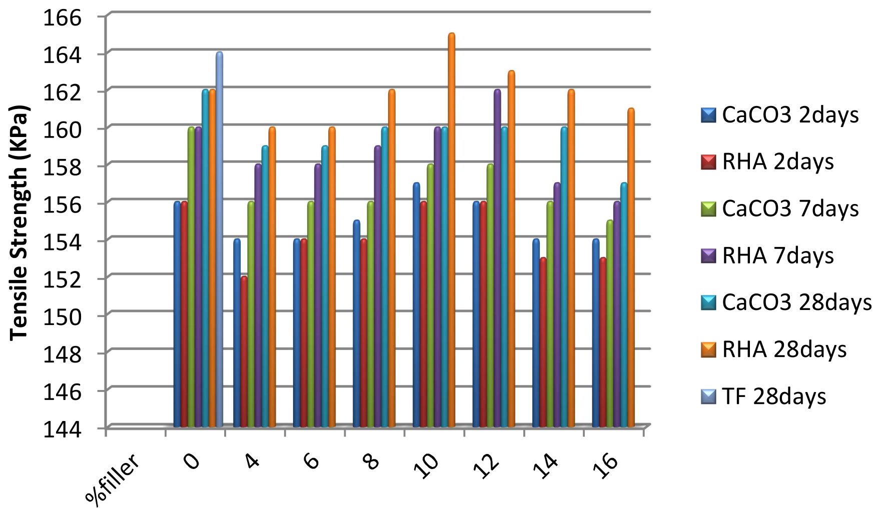

Fig.3: Plot of tensile strength versus weight% of $CaCO_3$ and RHA fillers in CTM

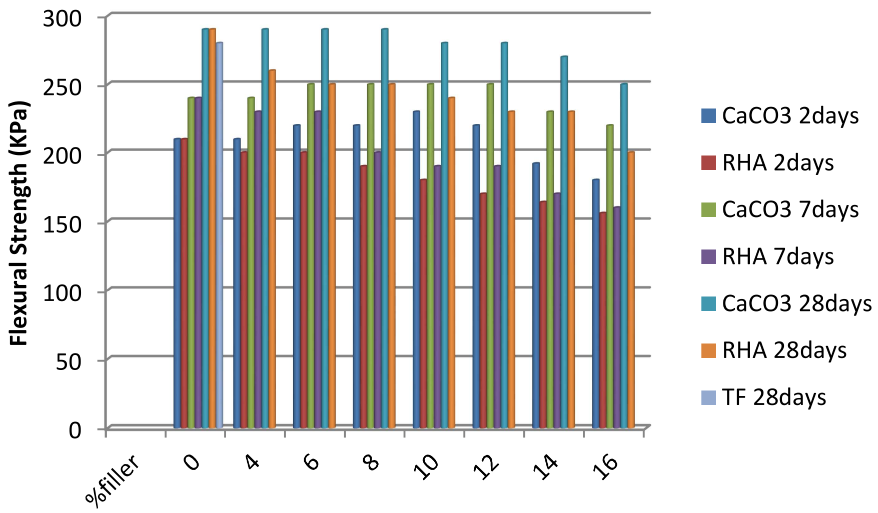

Fig.4: Plot of flexural strength versus weight% of $CaCO_3$ and RHA fillers in CTM

DISCUSSION

i. Bond Strength of Ceramic Tile Mortar

Table 3 and Fig. 1 show the values of the bond strength of $CaCO_3$-filled tile mortar and its RHA counterpart 2 days, 7 days and 28 days after application. After 2 days of application, it was observed that there was not much difference between the bond strength of $CaCO_3$-filled samples and RHA-filled samples. Remarkable difference in bond strength was shown 7 days after application; and this gap was further widened in the values recorded after 28 days of application. It was also evident that RHA-filled samples showed remarkably higher bond strength than $CaCO_3$-filled samples. In both samples, the bond strength increased from 4%w/w to between 10%w/w and 12%w/w of filler, and then started dropping from 14%w/w and above.

These observations suggest that after 2 days of application, the adhesive was still at the stage of water absorption by the substrate, water evaporation to the environment and hydration by the cement component. The pozzolanic effect of RHA on the bond strength came into play after this stage. This was reflected in the values in Table 3, Fig.1. The reduction in bond strength on the addition of 14%w/w and 16%w/w of filler was because the critical volume fraction

(CVF) of the filler/binder was exceeded. At this point, the interaction between the binder and the filler started diminishing. Improvement in shear bond strength of concrete by incorporation of RHA into the mortar has been reported by Nehdi et al (2003).

ii. Compressive Strength of Ceramic Tile Mortar

Table 4 and Fig. 2 show that the difference in the compressive strength between $CaCO_3$-filled tile mortar and RHA-filled tile mortar is in favour of RHA. The figures simply evaluate the response of the set mortar and, in extension, the tile plates to pressure and further compact form. The figures also indicated that the difference in compressive strength maintained a narrow margin even after 7 days of application. A wide difference was recorded after 28 days of application when 10%w/w and 12%w/w of RHA-filled samples recorded compressive strength of 12.9 MPa against 12 MPa and 12.4 MPa of $CaCO_3$-filled sample. Top Fix (the reference standard) in the same vein recorded 12.8 MPa. However, increase in filler proportion beyond 12% lowered the compressive strength of both RHA and $CaCO_3$-filled samples. This observed phenomenon suggests that in the formulation of rice husk ash based ceramic tile mortar, it is not advisable to add filler beyond 12 wt%.

The above observations agree with the earlier findings by Alessandra et al, 2007 and Scar, 2007, on bond strength that the nucleating effect of silica (the major constituent of RHA) helps in the formation of stronger bonds within the adhesivemortar and this also extends to the bond between the mortar and substrates. The delay before the marked difference equally shows that cementitious (pozzolanic) effect of RHA-filler on the mortar comes into play after some stage of setting has been attained. Also the difference in filler particle size might have helped to reduce the pore size of the RHA-filled samples and created a more compact system.

iii. Flexural Strength of Ceramic Tile Mortar

The values of flexural strength test of the various mixtures are shown in Table 5 and Fig 3. It was observed that the flexural strength of $CaCO_3$-filled samples increased even beyond the Top Fix values while RHA-filled samples showed the reverse. The increase in flexural strength of $CaCO_3$-filled sample was maintained up to 14%w/w filler. This trend of increase in flexural strength of $CaCO_3$ samples and the reverse in RHA-filled samples showed a wide margin from 2 days to 28 days after application.

The lower values observed in RHA-filled samples can be attributed to the fact that the positive contribution of RHA to bond strength makes the system stronger and stiffer than the $CaCO_3$ admixtures. Less number of void spaces in the case of RHA admixtures, as was reported in the compressive strength test, can also be one of the contributing factors to the lowering of flexural strength values of RHA admixtures.

iv. Tensile Strength of Ceramic Tile Mortar

Table 5 and Fig. 4 show the tensile strength of the various admixtures of RHA- and $CaCO_3$-filled mortar. The figures depict decrease in tensile strength of the mortar on the addition of 4%w/w of both $CaCO_3$ and RHA. This decrease was maintained up till the addition of 8%w/w of both filler. It was also observed that 2 days after application, RHA admixtures had lower tensile strength than the $CaCO_3$ admixtures. This trend was reversed 7 days after application and maintained 28 days after application when the RHA-filled sample recorded 0.165MPa against 0.164MPa of Top Fix. It was inferred that the stronger bond formation within the RHA admixture was responsible for the higher tensile strength recorded by RHA-filled mortar over $CaCO_3$-filled mortar.

CONCLUSION

In conclusion, the suitability of RHA as filler in ceramic tile mortar has been established in this investigation. When compared with $CaCO_3$ filler, RHA has higher degree of dispersion and better consistency in adhesives than $CaCO_3$. The cementitious (pozzolanic) property of RHA gives synergy to the binding property of the cement and RPPs. Thus, RHA-filled ceramic tile mortar samples have higher bond strength than the $CaCO_3$-filled samples. Also the compressive strength and tensile strength of RHA-filled ceramic tile mortar were higher than the $CaCO_3$-filled samples. The greater number of particles per unit volume of RHA in mortar (due to its low specific gravity and high surface/volume ratio) reduces void spaces in the mortar. The reduction in void spaces and probably the increase in bond strength reduce the flexibility of the RHA-filled ceramic tile mortar. This justifies the low flexural strength observed in the RHA-filled ceramic tile mortar. However, the values obtained in the determination of bond strength and mechanical properties of RHA-CTM in comparison with Top Fix reveal the closeness between the two.

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