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Article

Comparison of hemp fibres with macro synthetic fibres in lime-metakaolin matrix incorporating pumice as coarse aggregates

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ABSTRACT

Significant developments are observed in the design of composite building materials nowadays, especially on environmental and sustainability issues. For structural usage, nowadays researchers reveal lime, which is the traditional binding material known since ancient times. Lime known for its sustainable feature is often used with supplementary cementitious materials (SCM) such as metakaolin, fly ash, ground granulated blast furnace slag, and silica fume due to unfavourable properties regarding durability, strength, and slow hydration rate. On the other hand, cellulosic fibres with the advantages such as low density, high tensile strength, and moderate elastic modulus have cost competitiveness and eco-efficiency for fibre-reinforced composites. The structural use of cellulosic fibres may be possible if the degradation of hemp fibres in an alkali environment is mitigated. In this study, the experimental studies on hemp fibres were carried out by comparing with two types of macro synthetic fibres in a lime+metakaolin (L+MK)-based matrix. Durability as well as compressive and flexural characteristics were addressed in those fibrous matrices. First time in the current literature, macro synthetic fibres were included in the lime-based mixture. Besides, as a new contribution, coarse lightweight aggregates (LWA) were incorporated into this type of matrix with those fibres. The experimental findings indicate that the degradation of hemp fibres can be mitigated successfully, and three types of fibrous mixtures provide proper mechanical characteristics in their categories.

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INTRODUCTION

Lime is an important binding material from the past to the present and is a natural component for sustainable design. Cement came into prominence after the industrial revolution. However, due to the relatively high carbon footprint of cement, lime as a traditional material has become popular as a sustainable structural material again. On the other hand, eco-friendly and low-cost cellulosic fibres such as hemp, jute, and flax have become another important part of sustainable design with their proper mechanical characteristics (Yan et al., 2014) in recent years.

Air lime (L), which is different from natural hydraulic lime (NHL), should not be used as a binder alone due to

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poor durability and mechanical characteristics. Hence, supplementary cementitious materials (SCM) such as blast furnace slag (Seo et al., 2021), fly ash and silica fume (Koteng and Chen, 2015), and metakaolin (Silva et al., 2014) are combined with air lime for secondary hydration reactions.

Polymeric fibres such as polypropylene or nylon fibres have low elastic modulus and high elongation capacity (Zhang et al., 2020), and are used in implementations not requiring high toughness, often to prevent drying shrinkage cracks. Macro synthetic fibres are a rather new group of polymeric fibres and the studies in the literature focus on only cementitious matrices (Altoubat et al., 2009; Amin et al., 2017). On the other hand, cellulosic fibres with moderate elastic modulus are sustainable and cost-competitive materials. Using cellulosic fibres with structural aims is only possible by mitigating degradation under alkaline conditions. Lignin and hemicellulose content of cellulosic fibre can easily degrade (mineralisation, decay of cell wall) in an alkaline environment and the fibre loses its integrity (Wei & Meyer, 2014; Ardanuy et al., 2015). Removal of these unstable phases necessitates the fibre treatment through alkali solutions (e.g., NaOH), or more practically and economically SCM replacement to fix Ca(OH)2 (portlandite, CH). Alkali treatment may not be practical, economical, and effective (le Troëdec et al., 2009; Kabir et al., 2013). The other alternative via SCM for cellulosic fibre-reinforced concrete, especially silica fume and metakaolin mitigate the degradation in fibres, and prevents the drop in the mechanical characteristics (Mohr et al., 2007). For lime binders, some studies (Gameiro et al., 2012; Pavlík & Užáková, 2016) indicate the efficiency of metakaolin to remove CH. Metakaolin (MK) usage enables a more economical solution compared to silica fume as well.

Among different cellulosic fibres, hemp (*Cannabis Sativa L*) is cultivated for the manufacturing of ropes, textiles, and papers over the years. However, in the construction industry, mostly shiv (the woody core of the *hemp* plant) is used as coarse aggregate in low-strength lime concrete for insulation purposes (Delannoy et al., 2018). Hemp, as a fibre type, can be combined with cement-based (Sedan et al., 2008) or lime-based mortars (le Troëdec et al., 2011) as chopped fibres or textile mesh in last years. The purpose of this study is to enhance the structural performance of hemp fibre in a lime-based matrix and to compare it with macro synthetic fibre. The current literature on lime, metakaolin and cellulosic fibres especially is on mortars (Walker et al., 2014; Wang et al., 2022) and there are no studies on lime-based matrix reinforced with macro synthetic fibres, as the authors' knowledge so far. In addition, coarse lightweight aggregates are also a new contribution to current literature for this matrix type including cellulosic and macro synthetic fibres. The experimental findings indicate the hemp fibre and two types of macro synthetic fibres result in proper mechanical and durability characteristics in a lime+metakaolin-based matrix.

MATERIALS AND METHODS

Fibres

Two types of macro synthetic (Figure 1a, b) and cellulosic-based hemp fibre (Figure 1c) were used in the experiments. The main properties of those fibres are given in Table 1, the volumetric ratio in the mixture is V_f=1%. Hemp fibres were used without applying any chemical treatment and by

Table 1. Characteristic properties of fibres

	Copolymer Fibre* (PP/PE)	Polypropylene Fibre* (PP)	Hemp Fibre (H) (Thygesen, 2006; Dittenber & Gangarao, 2012)
Raw material	Pure Copolymer	Polypropylene	Hemp
Length (mm)	54	54	54
Tensile strength (MPa)	550–750	550	690
Modulus of elasticity (GPa)	5.75	8.5	30–`70
Density (g/cm^3)	0.91	0.91	1.5

*Manufacturer data.

Figure 1. Fibres types. a) Copolymer fibre, b) Polypropylene fibre, c) Hemp fibre.

cutting them to a length of 54 mm. Due to the high water absorption capacity of hemp fibres, up to five times their own weight (Elfordy et al., 2008), they were soaked in water for two hours prior to mixing. Then, their surfaces were wiped with a towel (Poletanovic et al., 2021), and then they were added to the mixture. Co-polymer fibres are in a twisted bundle form composed of thin filaments (<1 μm), PP fibres are in 0.5 mm × 1.5 mm textured multi-filament strip form as well. Each hemp fibre essentially consists of numerous individual fibre cells down to <0.1 μm in diameter.

Aggregates

Fine aggregates are natural silica sand (0.125–2 mm) and crushed limestone (max. 4 mm). As coarse aggregates, volcanic pumice (VPA) aggregates (D_{max}= 9 mm) from the Nevsehir region in Turkiye were used. Physical tests were carried out according to ASTM C127-15 (ASTM C127-15, 2016). Water absorptions in 10 min and 24 h were determined at 13.1 and 19.9% by weight, respectively. Loose, and rodded bulk densities, and saturated surface dry density were found as 0.65, 0.68 g/cm^3, and 1.27 g/cm^3, respectively. Pumice aggregates were soaked in water for 24h prior to the experiments. The volumetric ratios of coarse VPA, silica sand and crushed limestone were chosen as 50%, 30%, 20%, respectively. Sieve analyses of the aggregates, combined gradation, and comparison with the Fuller curve are given in Figure 2.

Lime-Based Matrix

Lime and metakaolin were used as binding materials, the chemical composition and the particle size fractions are given in Table 2 and Figure 3. In order to decide the quantities of L and MK as well as water to binder ratio, the lime-based mixtures in the literature were examined. The targets for compressive strength were realised to be very low (with shiv) (Kinnane et al., 2016), and low (Stefanidou et al., 2017). MK replacement was used as 1:1 to remove CH from previous literature (Silva et al., 2014).

Mixture Characteristics

For the usage with structural aims as well, to achieve a compressive strength about 20 MPa was adopted. As all the coarse aggregates are pumice aggregates a few preliminary trials on the mixtures were carried out, and finally, it was decided to use a binder content of 350 kg/m^3, and water to binder ratio of 0.45. The quantities of ingredients and fresh densities of the mixes are given in Table 3. Herein, LLM denotes the reference mix, the first letter (L) implies lightweight, and LM indicates lime+metakaolin. H, PP/PE, PP signify hemp, copolymer fibre and polypropylene fibre, respectively.

In the preparation stage of the mixture, first, dry materials (binders, aggregates, and fibres) were mixed, then the water divided into three equal parts was added to the dry mixture.

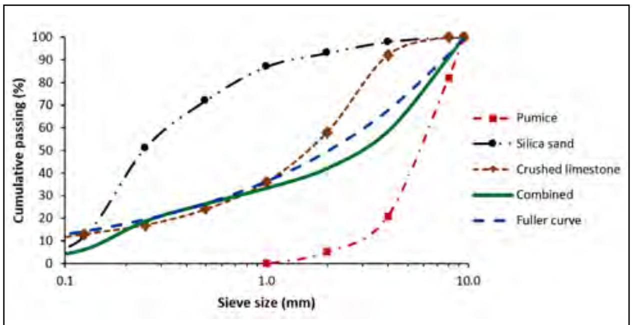

Figure 2. Sieve analyses of aggregates and combined grading.

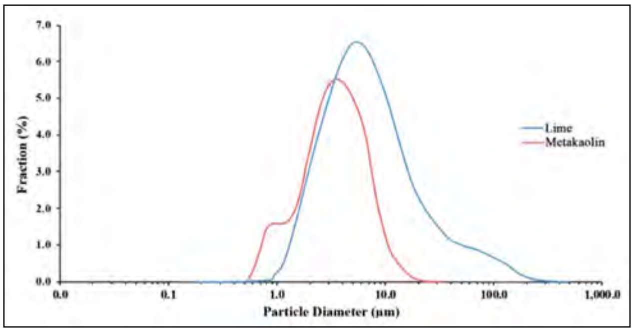

Figure 3. Particle size distribution of lime and metakaolin.

Table 2. Chemical composition (in wt.%) of lime and metakaolin

Binder materials and symbols	CaO %	SiO_2	$Al2O_3$	$Fe2O_3$	MgO	SO_3	Blaine fineness, m^2/kg
Lime	90	2.5	2.0	2.0	1.0	2.0	1072
Metakaolin	0.19	56.10	40.23	0.85	0.16	-	14600

Table 3. Mixture characteristics

Mixture	Lime (L)	Metakaolin (MK)	Pumice	Crushed limestone	Silica sand	Hemp fibre	Fresh densities
	kg/m_3					V_f (%)	kg/m^3
LLM	175	175	422	350	523	0	1790
LLM-H	175	175	415	345	514	1	1782
LLM-PP/PE	175	175	415	345	514	1	1799
LLM-PP	175	175	415	345	514	1	1792

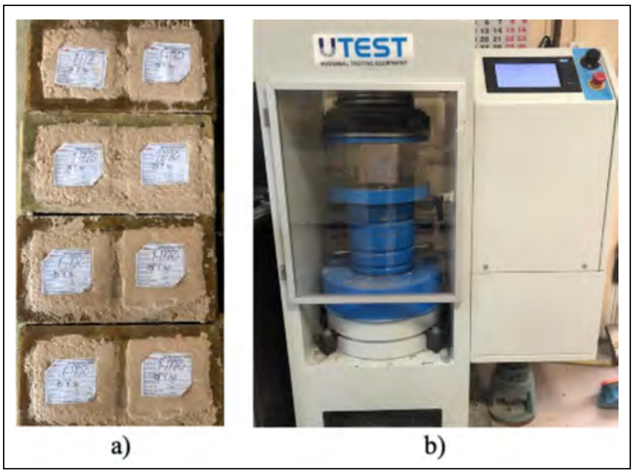

Figure 4. a) Cubic specimens, b) Compression test machine.

After then, about 1.5% HRWR was added in a controlled manner along with one-third of the water. The mixing process continued until the formation of a homogeneous mixture and a target slump of 10-15 cm was obtained. After demoulding one day later, the samples were cured at 20±2°C and 50%RH until 7- and 28- days for testing. In addition, the aging period following 28 days was conducted for the samples of LLM-PP/PE, LLM-PP, LLM-H series in a hot water tank at a constant temperature of (50±2)°C for 10 days according to ASTM C1560-03 (ASTM C1560-03, 2016).

Meanwhile, the fresh density for LLM samples was measured as an average 1790 kg/m^3 (±15 kg/m^3) (Table 3). Theoretical fresh density with normal aggregate (limestone, by assuming its particle density as 2700 kg/m^3) is calculated as 2285 kg/m^3, this means 22% lower density for LLM due to coarse pumice aggregates.

PREPARATION OF SPECIMENS AND TEST SET-UP

For compressive strength tests, three cubic specimens (100 mm) were prepared for 7 and 28 days in each series, i.e., total of 24 specimens. Those tests were performed in accordance with BS EN 196-1 (BS EN 196-1, 2016) (Figure 4a) using 3000-kN UTC Automatic Compression Testing Machine (Figure 4b).

For flexural tests, seven prismatic specimens (40 × 40 × 160 mm), were prepared (BS EN 12390-1, 2021) for 7, 28 days, and after aging in each series, i.e., 84 specimens (Figure 5a). Three-point bending tests were carried out in a deformation-controlled testing machine (MTS Criterion Model 43) at a load rate of 0.3 mm/min (BS EN 12390-5, 2019) (Figure 5b). Accelerated aging tests were conducted in accordance with ASTM C1560-03 (ASTM C1560-03, 2016).

A typical load-deflection diagram is given in Figure 4c. P_{cr} and Δ_{cr} in stage (I) signify critical load level and deflection that the matrix cracks. After a sudden decrease in Pcr level, the fibres bridge the matrix cracks and transfer loads. The other terms P_p and Δ_p in stage (II) define peak load and peak deflection. The following stage (III) is involved in toughness and Δ_u represents the ultimate deflection.

TEST RESULTS

Compressive Strength Tests

Compressive strengths of four series in 7 days and 28 days are shown in Figure 6 with standard deviations in parenthesis. For LLM-PP/PE and LLM-PP series with macro synthetic fibres, increments of 19% and 13% compared to 28-day reference specimens were observed, respectively. From 7 days to 28 days, the least change (46%) within all fibre-reinforced series was observed in LLM-PP/PE series.

In the hemp-fibre series (LLM-H), 7-day compressive strength decreased by 33% compared with reference ones, however, the reduction in 28 days was only 14%. The strength

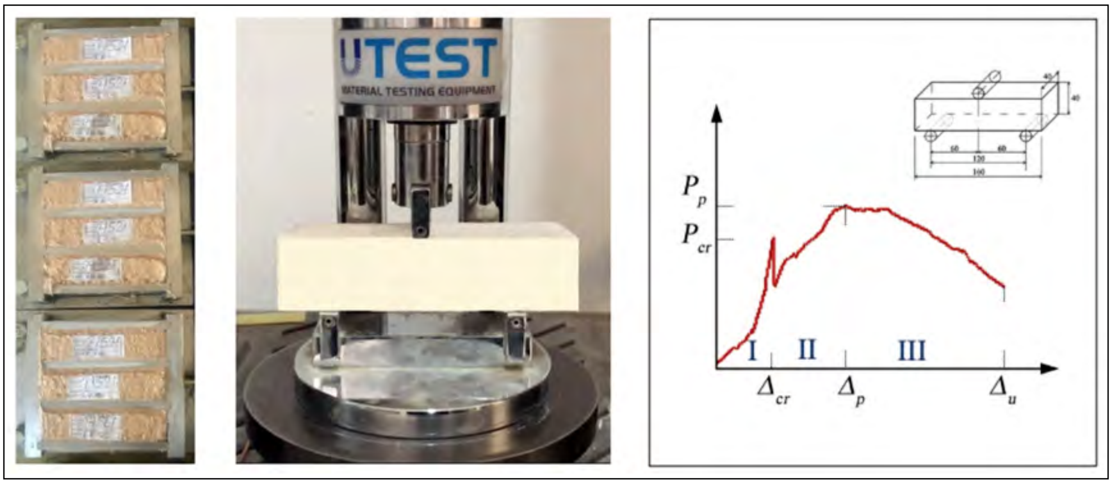

Figure 5. a) Prismatic specimens b) Bending test set-up c) Typical load-deflection diagram.

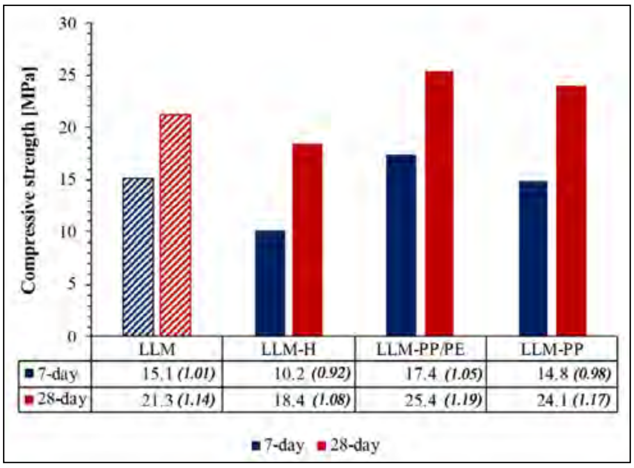

Figure 6. Compressive strengths of four series.

increment from 7 days to 28 days is 80%, the highest one within four mixtures. Herein, the higher reduction in 7 days can be partially attributed to the saturation of hemp fibres with water prior to the experiment. As another parameter affecting 7 and 28-day strengths, it is noted from a previous study (Awwad et al., 2012), the decrease in compressive strength is expected as the cellulosic fibres in the matrix are weak under compression.

Bending Tests and Evaluations

For all series, the flexural parameters (P_{cr}, P_p, Δ_p, Δ_u) in bending tests (Figure 5c), are given with averaged values for the tests of 7-, 28-day and after aging in Figures 7 and 8, the values in parenthesis are standard deviations.

The peak loads (Pp) in the series with macro synthetic fibre (LLM-PP, LLM-PP/PE) significantly increased (55–60%) in 28 days compared with reference samples (LLM). From 7 days to 28 days, the increment of P_p is the highest (64%) in the LLM-PP/PE series and the second highest (31%) in the LLM-PP series within four series. The ultimate deflections (Δ_u) enhanced about two times compared with the deflection in peak load (Δ_p). The highest increase (38%) in Δ_u values occurred in LLM-PP series after aging. Two polymeric macro synthetic series are essentially in hydrophobic characters (Bentur & Mindess, 2007). The flexural performances of those fibres in this study are attributed to better bonding performance owing to mechanical shear resistance due to their twisted bundle and textured multifilament forms in addition to interfacial frictional adherence. It is interesting that the frictional resistance governed the flexural behaviour, especially after aging, and this behaviour indicates the mechanical anchorage between hydrated products and fibre surface not to be so effective in this stage. In addition, there is an abrupt drop, or loss in bonding, immediately after the peak load in the LLM-PP series after aging.

In hemp fibre specimens (LLM-H), a 17% increment in P_p was observed from 7 days to 28 days. P_p values, 28 days and after aging, are 8%, 24% higher than P_{cr}, respectively. Ultimate deflection (Δ_u) increased 38% from 7 days to 28 days and decreased only 15% after aging. Deflection capacity over three times exists from Δ_p to Δ_u level, and there is no abrupt drop. The ductile behaviour even after aging is attributed to the mitigation of degradation due to binding

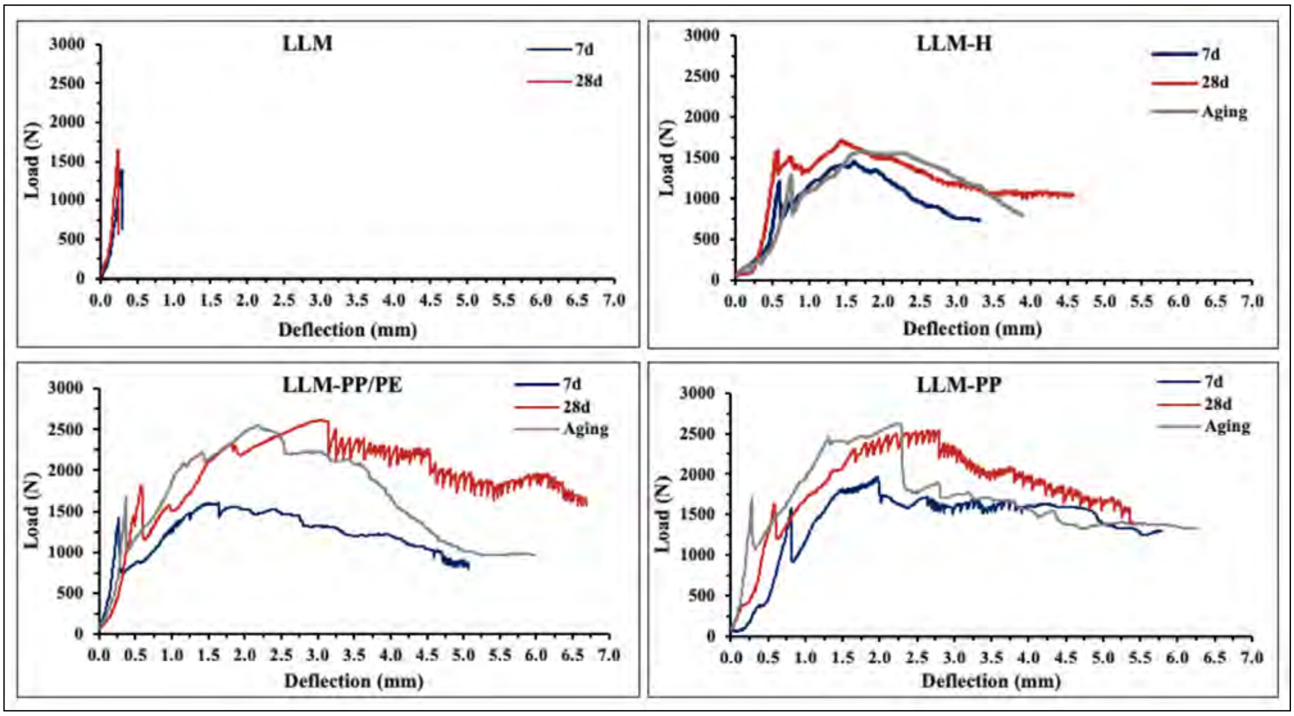

Figure 7. Load-deflection curves of mixes at 7, 28 days and after aging.

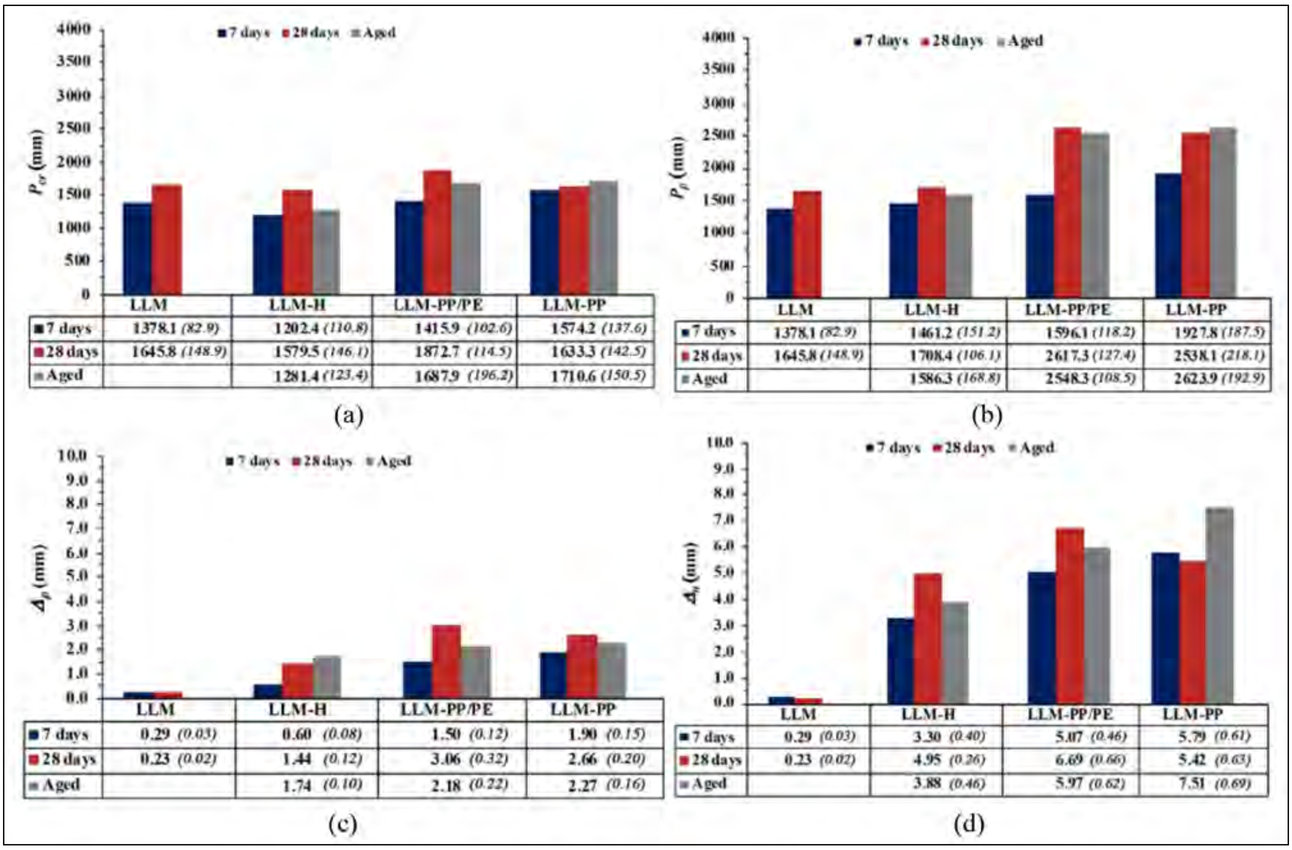

Figure 8. a) Critical load (P_{cr}) b) Peak load (P_p) c) Deflection in peak load (Δ_p) d) Ultimate deflection (Δ_u).

CH through MK substitute with air lime. Otherwise, the brittle behaviour via a sharp drop is expected after peak load, largely similar to the LLM series. P_p and Δ_u levels are not as much as macro synthetic fibres, which can be attributed to the fact that hemp fibres are cellulosic-based, hydrophilic, and in a different category.

DISCUSSION

Air lime which has non-hydraulic properties is known to be unsuitable especially in point of durability characteristics, e.g., low frost or moisture resistance, high shrinkage strains (Pavlík & Užáková, 2016). In order to gain hydraulic characteristics as well as higher strengths, the L+MK combination is known to be very effective. There are several compounds from that chemical reaction, one of those products is katoite ($Ca_3Al_2(SiO_4)(OH)_8$) having negative effects on the durability of the matrix. A 50% MK substitute was proven to the katoite formation to prevent during 1.5-year curing (Silva et al., 2014), and this ratio – not a lower ratio – fixes almost CH starting from the early period of curing as well.

The modulus of elasticity in polymeric fibres is low, and the deformation capacity is high (up to 30%) (Zhang et al., 2020) compared to well-known fibres such as glass or basalt fibres (2–3.5%). In this study, the deflections in the series with macro synthetic fibres (Figures 7 and 8, Figure 9) are attributed to those characteristics. On the other hand, some cellulosic fibres such as hemp, jute, and flax attract attention with their moderate modulus of elasticity, and deformation capability similar to glass or basalt fibres (Yan et al., 2014; Zhang et al., 2020). Finally, rather similar tensile strengths to polymeric fibres are also an important feature for the structural use of cellulosic fibres. Thus, the lower deformation ability of hemp fibres in bending tests is not a deficiency, similar deflections are observed in glass and basalt fibres for moderate toughness.

The hydrophobic structures of polymeric fibres (Bentur & Mindess, 2007) negatively affect interfacial bonding with cement. In the absence of chemical reactions, the adherence can be tolerated to some extent by enhancing mechanical bonding, e.g., the changes in the form such as textured multifilament or twisted bundle similar to macro synthetic fibres in this study. Thus, the pull-out of macro synthetic fibres was substantially observed through debonding in Stage III (Figures 5c and 7), especially in 28 days, and this performance is very satisfactory. However, it is observed that the mechanical anchorage in 7 days and after aging was not so effective. Herein, MK ratios lower than 50% may be proposed to explore the variation in flexural performance, e.g., 33% MK ratio in which CH is not fully consumed and katoite is not formed in the long term (Silva et al., 2014).

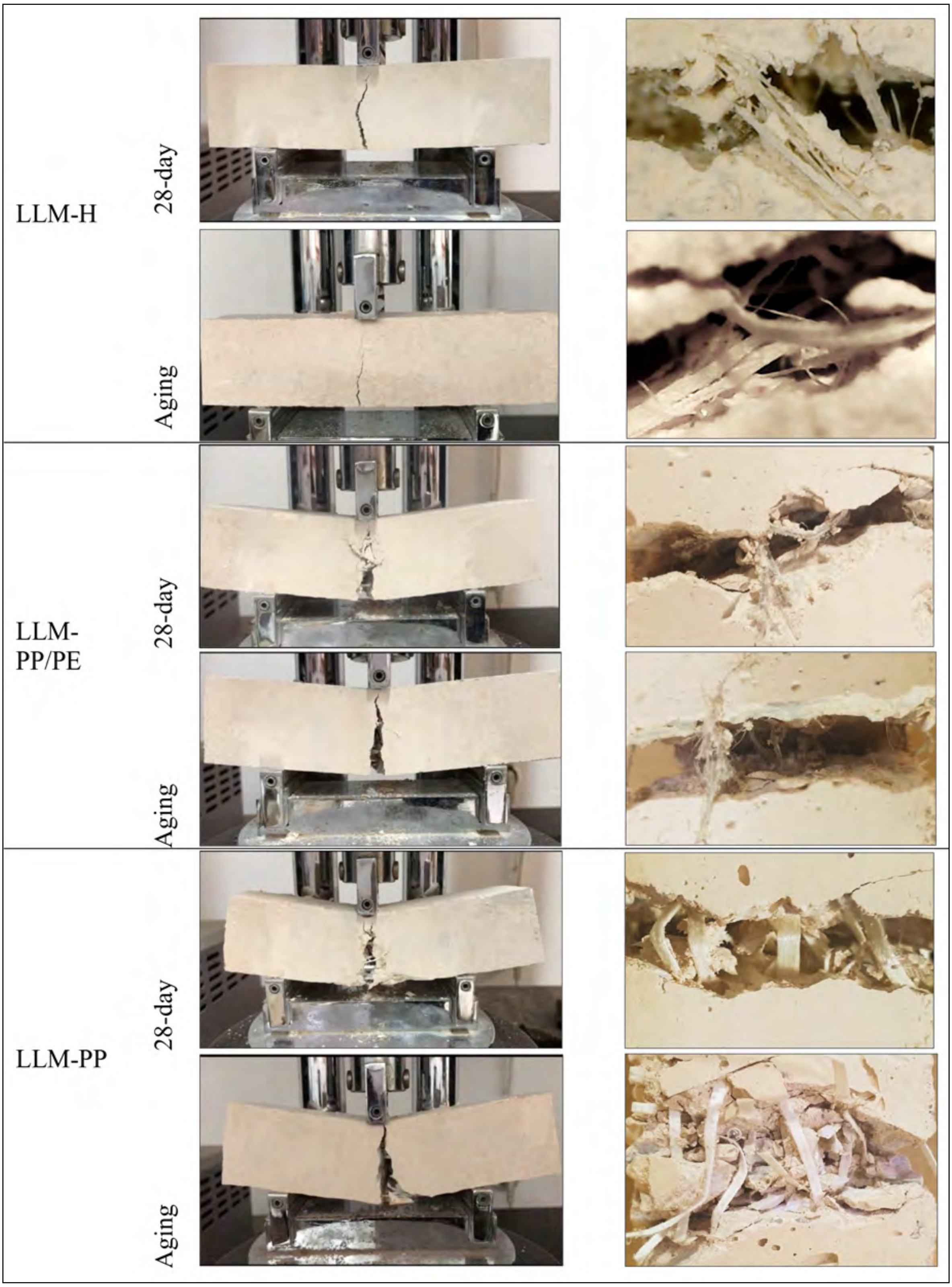

Figure 9. Crack patterns and microscopic images in 28-day and after aging.

Brittle behaviour in mixtures with cellulosic fibre is attributed mainly to the crystallisation of CH in the lumen, walls, and voids in the cellulosic fibre (Ardanuy et al., 2015). The adsorption of calcium and hydroxyl ions (Filho et al., 2013) by fibre surface leads to the disintegration of fibre structure (Ghosn et al., 2020), and the deformation loss

Figure 10. Views from hemp fibres after aging.

under bending happens through a sudden rupture almost without pull-out even at 28 days. In this study, especially the mitigation of the degradation and the maintenance of flexural deformations were the main targets. Enhanced flexural performance rising from the experiments is a result of consuming CH in the matrix, and brittle behaviour was prevented. Its evidence can be observed in the existence of strain softening (Stage III, Figure 5c) governing the toughness (Figure 7), crack mouth (Figure 9) and clean fibre surfaces (Figure 10).

As another comparison, the fresh density of LLM cubic samples was measured as an average of 1790 kg/m^3 (Table 3), which is 22% lower than the theoretical normal-aggregate mix of 2285 kg/m^3 (crushed limestone by assuming as 2700 kg/m^3). Prior to compressive strength tests, the density of LLM samples was measured at 6.3% lower (average 1678 kg/m^3) as a result of coarse pumice aggregates in the mix design which have lower density and gradually release water for internal curing.

The combination of L+MK, in comparison with the poor performance of L, is very efficient to decrease shrinkage deformations. Pavlík and Užáková (2016) investigated the effect of curing conditions and indicated that the 1:1 L+MK combination almost prevents the shrinkage deformations at sealed a chamber (RH 100%), and highly prevents it for RH 65%. For hemp fibre reinforced L+MK mortar (66%L+33%MK), drying shrinkage deformations decrease by about 60% (Wang et al., 2022) compared with lime mortar, smaller pore sizes and the enhancement in durability issues are other positive results. It is noted that the positive effect of coarse pumice aggregates (Akcay & Tasdemir, 2009) in cementitious binders to prevent the shrinkage cracks by gradual water release may be considered for fibre-reinforced L+MK composite in this study as well.

The fire resistance of fibre-reinforced lime-based composite is another issue to predict, and it depends on the ingredients. For lime- and blended mixtures (Pachta et al., 2018) L-mix has the lowest fire resistance and pozzolanic components significantly increase the fire resistance. By comparing with concrete, for a one-hour fire duration at 800°C (Hossain & Lachemi, 2007), the samples with coarse pumice aggregates maintain about 40% of the initial strength, that ratio is about 20% for normal-aggregate concrete; the melting of the PP fibres is below 400°C, however, some of the hemp fibres still present (Netinger Grubeša et al., 2018).

CONCLUSIONS

More importance should be given to sustainable design in buildings to reduce carbon footprint. In this study, the possibilities for more usage of sustainable resources in structural members have been explored. Lime and pumice aggregates were addressed as sustainable resources. Cellulosic fibres and macro synthetic fibres in lime-metakaolin-based matrix were studied experimentally. The coarse pumice lightweight aggregates were used in a lime-based matrix with those fibres. The results from this study are summarised below.

- In specimens with macro synthetic fibres, rather similar performances was observed. In 28-day specimens, a good mechanical adherence in the interface of hydrophobic polymeric fibres and matrix were observed, and debonding behaviour is dominant around peak load and beyond it. After aging, more smooth form of the P-Δ curve indicates the decline of mechanical anchorage and pull-out mode transforming to fibre slippage. The lime+metakaolin-based matrix positively affected the debonding behaviour of macro-synthetic fibres (Figure 7).
- CH removal through 50% metakaolin addition provides the pull-out mechanism in hemp-fibre reinforced specimens to maintain even after aging test which provokes CH release. The rather clean surfaces of the hemp fibres were also observed in optical micro photos (Figure 10). The mitigation of the degradation in cellulosic fibres was satisfied for hemp fibres successfully. There is an increase in deflection capacity over three times from Δ_p to Δ_u (Figure 8).
- Strain softening (Stage III, Figures 5c and 7) was observed in three fibre types as well. This stage points

out the toughness, and three fibre types seem to have suitable flexural performance in their own categories.

- As some suggestions; the hemp fibres can be alternative to polymeric fibres to prevent shrinkage cracks in mortar-based plasters, and the combination with coarse pumice aggregates may be used in RC slabs, composite decks of steel structures or timber structures to prevent drying shrinkage cracks for sustainable solutions. Pumice aggregates will also allow a more lightweight design and fire resistance.

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