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LIME-HEMP CONCRETE (LHC) ENHANCEMENT USING MAGNESIUM BASED BINDERS

M. Sinka*, G. Sahmenko, A. Korjakins, D. Bajare

Riga Technical university, Faculty of Civil Engineering, Institute of Materials and Structures,
Kalku str. 1, Riga, LV-1658, Latvia

*Corresponding author: e-mail: maris.sinka@rtu.lv

Abstract

The major CO_2 emitting countries have concluded an agreement aimed at reducing the CO_2 emissions at the Paris Climate Conference 2015. However, the emission levels are still on the rise. As the building material industry is one of the largest producers of CO_2, it should find solutions to limit these emissions, such as development of new materials with negative CO_2 balance and low thermal conductivity for energy saving during lifespan. The lime-hemp concrete (LHC) could be one of the solutions as it has negative CO_2 emissions balance (up to 80 kg/m^2) and low thermal conductivity (0,07-0,09 W/m*K). Even though, the LHC has been gaining popularity and recognition over the past years, it is still not used enough to contribute significantly to lowering the global CO_2 emissions. One of the reasons is the low mechanical strength of this material that can be mostly used only with supportive load bearing frame appropriate for the low rise buildings. It could be improved by enhancing the binder strength either through supplementing or substituting it with the magnesium based binders. Made of calcined MgO with possible additions of magnesium salts or other hardeners, the early and overall compressive strength shown by these binders is greater compared to hydraulic lime binders used in the LHC. The magnesium based binders also remain unaffected by the organic water soluble constituents that react with calcium ions in hydraulic lime, thus delaying or preventing hydration process. Various MgO binders are tested in this research, focusing mainly on mechanical strength and thermal conductivity, in order to understand the effect that adding the magnesium binders has on important LHC properties. The obtained results suggest that the MgO binders are viable alternative to hydraulic lime as their superior compressive strength allow for lower amount of binder to be added thus enhancing thermal conductivity or giving material wider possible use due to this strength increase.

Keywords:
Lime-hemp concrete, LHC, hemp, magnesium oxychloride, MOC, MPC

1 INTRODUCTION

In recent years global community has signed several agreements on reduction of the CO_2 emissions in the atmosphere, such as the Paris Agreement signed in 2015 and ratified by the EU in 2016 [Liobikiene 2017]. The EU also tries to limit its own emissions, for instance, with the directive EU2010/31/EU, that should reduce the CO_2 level by 20% by 2020 [Sinka 2015], Energy and Climate framework 2030 with aim to reduce greenhouse gases by 40% and improve energy efficiency by 27% by 2030 or Roadmap for moving to a low-carbon economy in 2050 with aim to reduce CO_2 emissions by 80% compared to 1990 [Liobikiene 2017]. Despite all this effort, the global CO_2 levels are still on the rise and in 2016 have permanently exceeded 400 ppm [Betts 2016]. To achieve the goals set by global community and the EU, a much wider scope of action is necessary.

As the construction material industry and the primary energy for households are among the biggest CO_2 emitters [Li 2017, Lin 2015], it is necessary to develop building materials that could address these industry problems. One of such materials is the lime-hemp concrete (LHC). It has several positive properties such as excellent thermal performance – high thermal capacity and low thermal conductivity [Walker 2014], high moisture buffering capacity and moisture transfer [Maalouf 2014] [Rahim 2015], and also high sequestered CO_2 amount [Ip 2012][Shea 2012].

Weakness of the LHC is its relatively low mechanical strength, allowing its main use only with supportive load bearing frame thus making it appropriate mostly for the low rise buildings [Latif 2014]. The other problem of the LHC is that lignin, sugars and other bio-based compounds retard hydraulic lime binder resulting in reduced early strength [Balciunas 2015].

These two problems can be addressed by enhancing the lime binder strength either by supplementing or substituting it with magnesium based binders. Magnesium binders have both – good compatibility with the bio-based aggregates and high early strength [Ma 2017].

Magnesium binders are made of magnesium oxide - MgO, produced by calcination of magnesium carbonate or magnesium hydroxide. Depending on the calcination temperature there are two types: caustic calcined magnesia which is highly reactive and obtained in low temperatures (700-800 ^{0}C) [Ruan 2016] and dead-burned magnesia with lower surface area and limited reactivity, made in high temperatures (1300-1500 ^{0}C) [Ma 2014]. Although these magnesium oxides have different reactivity and are usually supplemented with various additives, both types of magnesia are used within scope of this research.

Caustic magnesia can be made in binder by two approaches –carbonization or by using magnesium salts $MgCl_2$ and $MgSO_4$.

Carbonization is achieved by hydration of MgO that produces brucite ($Mg(OH)_2$) which in turn reacts with CO_2 and forms hydromagnesite ($4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$), dypingite ($4MgCO_3 \cdot Mg(OH)_2 \cdot 5H_2O$) and nesquehonite ($MgCO_3 \cdot 3_H2O$) [Unluer 2014]. This is the magnesium binder with the lowest CO_2 emissions in terms of production as all the carbon released in calcination of magnesium is reabsorbed [Pu 2016] and no high CO_2 emitting additives are used. Also it

has lower calcination temperature of 700-800 ^{0}C compared to that of lime at 900-1000 ^{0}C or Portland cement at 1450 ^{0}C [Pu 2016]. It can also be synthesized of seawater as waste from the desalination process [Dung 2016]. The problem with this type of binder is the slow strength gain as in accelerated conditions it can reach 40 MPa compressive strength in 28 days, compared to 8 MPa without acceleration [Dung 2016]. Due to its chemical compatibility with lime, it can be used as substitute as well as supplement for the lime binder.

Magnesium oxychloride (MOC) and oxysulfate cements also known as the Sorel cement were introduced shortly after discovery of the Portland cement [Li 2013]. It has high early strength, compatibility with various organic aggregates [Zhou 2012] and high fire resistance [Chau 2009]. In reacting with MgCl the magnesium oxide creates four main reaction crystal phases (phase 3, phase 5, phase 7 and phase 9). Two of these phases can exist stably in temperature below 100 ^{0}C, namely phase 3 ($3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$) and phase 5 ($5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$) [Xu 2016]. In specific conditions it can reach compressive strength up to 120 [Li 2013] to 140 [Xu 2016] MPa. As it also uses low temperature calcined magnesium it has lower CO_2 emissions, however only small portion of binder carbonates over time [Power 2017] therefore it cannot reabsorb all of the CO2 emitted during calcination. It is also non-toxic to humans as is widely used as orthopedic biomaterial [Tan 2014].

Tab. 1: Composition and properties of used raw materials

Name	Classification	MgO	CaO	SiO_2	Fe_2O_3	Al_2O_3	Size distribution	Poured density
RKMH-F	Caustic magnesia	73,0%	4,0%	4,0%	3,0%	1,0%	90% < 30 µm	0,8 g/cm^3
M-76	Dead-burned magnesia	81,0%	11,0%	2,0%	8,0%	0,3%	0 to 0,2 mm	-
MK	Metakaolin	0,1%	0,1%	51,8%	0,5%	34,2%	-	-

Name	Fibre	>20mm	10-20mm	0,63-10mm	Dust	Density	Thermal cond.	Moisture
Hemp shives	1,7%	0,5%	3,7%	92,0%	2,2%	108,36 kg/m^3	58,07 W/m*K	11,75%

Dead-burned magnesia can be used as binder with addition of monopotassium phosphate (KH_2PO_4), thus producing magnesium phosphate cement (MPC) as a type of Chemically Bounded ceramics. The reaction of MgO and monopotassium phosphate forms crystalline structure $KMgPO_4 \cdot 6H_2O$ which is named K-struvite or ceramicrete [Valle-Zermeño 2016]. It has high compressive strength of 80 MPa and more [Zhang 2017], as well as very fast setting time that can lead up to 80% compressive strength in 3h compared to 28 days [Ma 2017]. It can also be used with different organic aggregates [Donahue 2010] to create wall panels [Amiandamhen 2016], and with porous organic aggregates (rape stalk and hemp shives) to create insulation panels. [Ning 2015][Valle-Zermeño 2016]

In order to test various magnesium binders, their compatibility with hemp filler and to measure their

capacity to address the low strength and bio-incompatibility problems, various mixtures of magnesium mortar, the LHC and the MHC were made, substituting and replacing lime and hydraulic lime binders.

2 MATERIALS

2.1 Magnesium oxide

Magnesium oxide is made by calcination of either magnesium carbonate or magnesium hydroxide. Magnesium hydroxide is usually obtained by treating sea brines with lime, magnesium carbonate is mainly found in anhydrous form titled magnesite, and is obtained by mining. The reactivity of MgO mostly depends on calcination temperature and the two types of oxide can be separated.

Within scope of this research both types of magnesium oxides were used – caustic and dead-burned magnesia, both made by calcination of magnesite ($MgCO_3$) and produced in Europe. The dead-burned magnesia M-76 comes from Slovakian company Integra Ltd. and has been calcined in temperatures up to 1700 ^{0}C. Caustic magnesia CCM RKMH-F comes from Austrian company RHI AG. Their composition is presented in Table 1.

2.2 Lime, metakaolin and magnesium additives

The metakaolin for this research is obtained as waste by-product of porous glass granulate production factory in Lithuania "Stiklaporas" UAB. It is produced by use of kaolin clay as an anti-agglomeration agent for the porous glass granulate burned at 850 ^{0}C for 40-50 min. According to the SEM and the XRD analysis the obtained metakaolin is very similar to commercially produced making it appropriate for products that aim for reducing their environmental impact. Specific surface of the obtained metakaolin is 15,86 m^2/g. [Bumanis 2017]

Three types of lime are used in the research. Hydrated lime CL90 provided by Lhoist Poland Ltd. (CL), experimental formulated hydraulic lime containing 60% CL and 40% MK (FHL), and hydraulic lime binder used commercially for the LHC construction, containing 70% hydrated lime, 20% hydraulic lime and 10% additives (HL).

Tab. 2: Mixtures of samples

No.	Type	Shives, g	Water, g	MgO, g	Water extra,g	Ca(OH)₂, g	Metak., g	HL, g	MgCl, g	KPO, g
1	P1	1200	1500	1800	1440	-	-	-	-	1440
2	P2	1200	1500	1200	960	-	-	-	-	960
3	P3	1200	1500	600	480	-	-	-	-	480
4	P4	1200	1500	400	320	-	-	-	-	320
5	OX1	1200	1500	1800	-	-	-	-	1200	-
6	OX2	1200	1500	1260	-	-	-	-	840	-
7	OX3	1200	1500	900	-	-	-	-	600	-
8	OX4	1200	1500	600	400	-	-	-	400	-
9	FHL(M)1	1200	1500	1200	1200	720	480	-	-	-
10	FHL(M)2	1200	1500	600	600	360	240	-	-	-
11	FHL(M)3	1200	1500	450	450	270	180	-	-	-
12	CL(M)1	1200	1500	1200	1200	1200	-	-	-	-
13	CL(M)2	1200	1500	600	600	600	-	-	-	-
14	CL(M)3	1200	1500	450	450	450	-	-	-	-
15	HL	1200	1500	-	1200	-	-	2400	-	-
16	HL(M)	1200	1500	1200	1200	-	-	1200	-	-
17	FHL	1200	1500	-	1200	1440	960	-	-	-
18	MG	1200	1500	2400	1200	-	-	-	-	-
19	CL	1200	1500	-	1200	2400	-	-	-	-

For dead-burned magnesia a monopotassium phosphate supplied by Prayon S.A. is used, with P_2O_5 content at least 51,6%. The used magnesium chloride is magnesium chloride hexahydrate produced in Germany.

2.3 Hemp shives

Hemp shives are provided by local hemp producer "z/s "Rudeņi"". Properties of hemp shives are presented in Table 1. In previous research these hemp shives have proven to provide one of the best granulometric properties in achieving both – good thermal conductivity and high compressive strength [Sinka 2015].

3 METHODS

3.1 Mixing

As the shives are hydrophilic and should be premixed with water, mixing of biocomposites was done mechanically, adding water in two stages. The rest of water was added after the binder. The mix proportions according to Table 2. Binder with additives was premixed in dry state before adding it to shives.

The mixing was done in forced action double shaft laboratory mixer BHS DKX 0.06, at a speed of 60 rpm. The shives were added beforehand in all samples, mixer turned on, amount of water necessary for shives added by spraying it evenly over the shives and continued mixing for 1 minute. The binder in dry state was then added and dispersed evenly over the shives, so it stuck to the wet shives, afterwards adding the rest of water or $MgCl_2$ solution and mixing for 2 minutes.

The samples for phosphate biocomposite were made in similar manner as mentioned previously. A constant amount of shives was premixed with water. Magnesium oxide and potassium phosphate were added and mixed for 1 minute, only then the rest of water was added and mixed for 1 minute and then the moulds were filled.

The specimens for mortar test were made at the same binder/hardener/water ratios as indicated in Table 2, sand to binder ratio was 2:1.

3.2 Curing conditions

After mixing the samples were moulded in custom sized plywood moulds. Demoulding was done after 2 days. Afterwards the samples have been cured in laboratory conditions (20±2 ^{0}C and 40±10 %RH) for approximately 28 days, until weight equilibrium was achieved. Mortar samples were demoulded after 2 days and were kept until tests in laboratory conditions the same as the biocomposite samples.

3.3 Testing

The biocomposites were tested on thermal conductivity, as well as on compressive and flexural strength. Thermal conductivity was measured with LaserComp FOX600 heat flow meter, according to the standard LVS EN 12667 guidelines, settings of the test - 0 ^{0}C upper and 20 ^{0}C lower plate. The samples for compressive and flexural tests were produced by sawing the larger samples into 100x100xheight for compressive and 150x350xheight for flexural strength. The tests were done on Zwick Z100 universal testing machine. The pressure was applied at speed of 10mm/min, recording the force-deformation diagram during the process. The test part on compressive strength was performed until 10% relative deformations (according to the LVS EN 826), flexural – until rupture.

4 RESULTS AND DISCUSSION

Results from mortar and biocomposite compressive strength tests are presented in Fig. 1. The samples of density 350 +/- 10 kg/m^3 were chosen for biocomposites, a standard 2:1 sand/binder ratio was used for mortar.
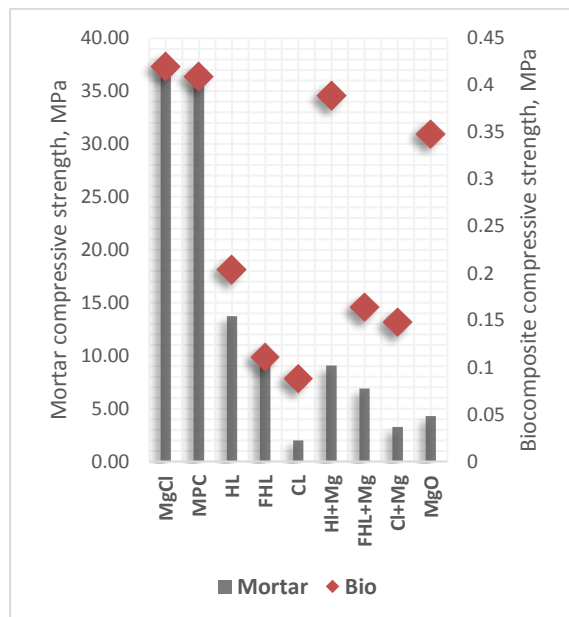

Fig. 1: Mortar vs biocomposite strength

As can be seen MOC and MPC binders have overall the highest compressive strength as expected from the literature sources. However only one third of compressive strength was reached compared to that can be achieved with oxychloride or orthophosphate cements, most probably due to low class of MgO binders that have lower amount of free MgO. As it is already known from previous tests – commercially available HL and experimental FHL have compressive strength of approximately 13 and 10 MPa as a mortar, pure air lime CL90 – just 2 MPa.

Adding of MgO to hydraulic lime binders lowers their strength, as seen in Fig. 1, due to lower amount of fast forming calcium silicate phases. Moreover, the early strength at 14 days was even lower, the MgO supplemented hydraulic limes had 50% pure hydraulic lime strength. The strength of pure MgO is higher than CL90 as brucite (Mg(OH)2) is formed of MgO before it can fully carbonate, and provides considerably

Ca(OH)$_2$. It is shown in other studies that upon fully carbonating in artificially created conditions, compressive strength of Mg based binder can reach up to 40 MPa [Dung 2016], but carbonation for Mg in specific relatively dry conditions takes considerably longer, that is reflected through achieved compressive strength of only 4,30 MPa.

In Fig. 1 it can also be seen that although MOC and MPC mortars have 3 times higher strength compared to other binders, this gap is considerably more narrow when they are used as binders for bio-composites – 0,42 and 0,409 MPa for oxychloride and phosphate binders, followed by pure MgO binder and HL(M) binder – 0,348 and 0,389 MPa respectively. It can also be noted from this graph that the CL90 based binders FHL and CL have the lowest strength as a biocomposite, although the strength of mortar was relatively high – 9,41 MPa. As found in previous studies [Sinka 2015] these binders have lower early strength and lower compatibility with biocomposites due to highly alkaline environment which enables additional amount of set retarding that can been noted by unhardened binder in the middle of the sample and by hardened and much stronger crust.

Addition of 50% MgO to all lime binders had a positive effect on their compressive strength (Fig. 1 and Fig. 2). The increase was 90% for HL, 48% for FHL and 68% for CL. It can be seen from the graph that the MgO binder has the highest ratio of strength increase compared to mortar with biocomposite. This is due to lower alkalinity of MgO and overall biocompatibility resulting in considerably higher strength for biocomposite than could be expected from mortar of 4,30 MPa strength. This good compatibility also allows increase in strength of the supplemented lime based binders, although the CL90 lime based ones exhibit lower strength increase than the HL based binder. Moreover, data in Fig. 3 of compressive strength crosswise to compaction direction shows that the HL with MgO addition exhibits the lowest strength increase, just approximately 25% of the CL based binder increase. This, in fact leads to conclude that differences in strength increase with MgO addition were based on technological process, and that all the lime binders exhibit similar increase.

The difference of thermal conductivity between lime based binders and the binders with added MgO can be seen in Fig. 4, it is below 0,0025 W/m*K for all mixtures. Although this difference is meager in material scale, it can have greater impact when calculated, for example, in a whole building scale. This difference could be linked to the difference in compressive strength as was previously found [Sinka 2015] and due to this the thermal conductivity for composites with partly unhardened inner structure was lower.
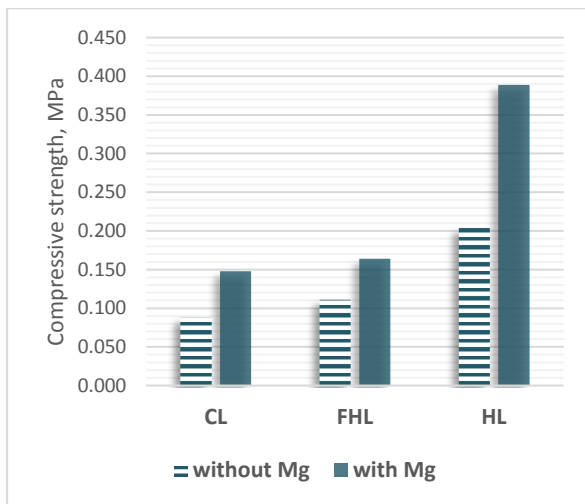
Fig. 2: Compressive strength of biocomposites with lime based binders
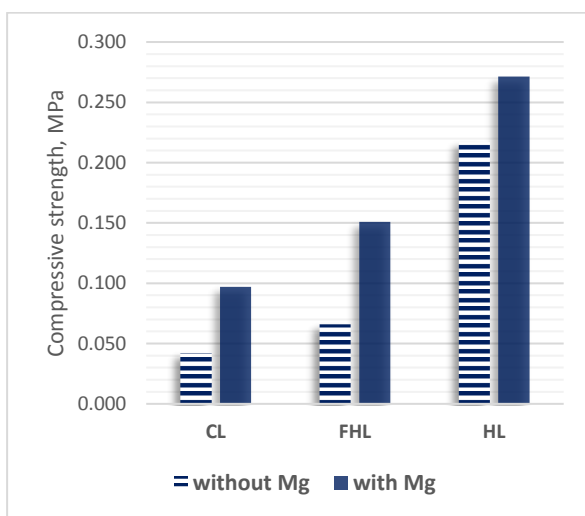

Fig. 3: Compressive strength crosswise of biocomposites with lime based binders
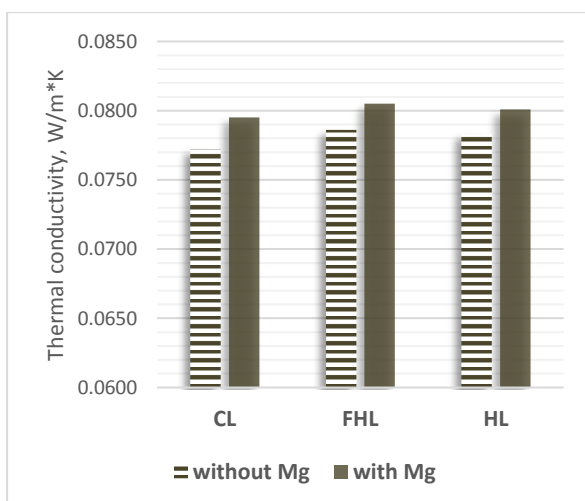

Fig. 4: Thermal conductivity of biocomposites with lime based binders

As seen in Fig.5 the trend of higher strength binder and higher thermal conductivity applies only to MOC binder, as MPC have lower thermal conductivity despite the high strength. However the MOC's higher λ can be explained according to the previous paragraph, as MPC has lower λ because surface area of the dead-burnt magnesia has positive

influence on thermal conductivity as the binder volume is lower than in similarly dense composites. From the results it can be seen that they correspond to the data obtained from research and provided by manufacturers, for example a 350 kg/m^3 density with 0,080 w/m*K [Sinka 2015], but this refers only to the samples without any extra pressure applied. Compressing the samples is one way to achieve higher density without thermal conductivity loss, because smaller amount of binder is necessary and they correspond with data from different packing of the inter-shive voids [Sinka 2015]. As shown, the line is linear up to approximately 450-500 kg/m^3. Further as the amount of shives reaches less than 25% of binder mass, the graph lines become more curved and thermal conductivity increases more rapidly compared to density, although such densities are not relevant in scope of self-bearing wall insulation materials.

The results of compressive strength and density ratio can be seen in Fig. 6. It can be observed that MOC and MPC binder biocomposites reach higher strength compared to lime based biocomposites in all of the measured densities, the lime based being approximately 3 times weaker at low densities, and presenting even wider gap after reaching 350 kg/m^3. The difference of compressive strength was expected and can be attributed to higher overall and early strength of MOC and MPC binders as well as to biocompatibility of MgO. After reaching 350 kg/m^3, curves of the MOC and MPC graphs become steeper, which can be explained by the fact that the amount of binder allows it to work in a uniform grid structure and properties of the material become more similar to those of the binder.

Adding of magnesium based binders to the LHC has proven overall to be a valuable trial. Strength has increased in all samples with added MgO, without any significant side effects, mainly because of bio-compatibility of MgO. It was possible to achieve significantly lower densities (250 kg/m^3) for composites with compressive strength adequate for self-bearing wall insulation material (around 0, 2 MPa). In addition, these binders have high potential to be used for prefabricated block or slab production as they have better strength/thermal conductivity ratio.

As the possible downsides/unclear areas of the material use were noted the following:

1. The reached strength permits reduction of the binder amount for MOC and MPC to 70% of shive weight, which in turn could mean possible negative impact on various properties that rely on dense binder coverage, such as fire and bio resistance.
2. One of the major advantages of the LHC is its low environmental impact, particularly the negative CO_2 emissions due to lime carbonization and hemp growth [Ip 2012][Shea 2012]. Alternative binders, which are not using carbonization to harden, cause partial loss of this CO_2 sequestration. The environmental impact of magnesium oxide and its hardeners (specifically the KPO) should also be calculated, as well as that of increased transport loads due to more limited availability of magnesium in comparison to lime.
3. The effect of water and water vapor on MOC and MPC biocomposites should also be evaluated. As

MOC is known for its instability in wet environment, necessary protection measures should be researched. MPC in turn is a water resistant binder, leading to inquiry about MPC's biocomposite water vapor permeability, which is one of the key advantages the LHC.
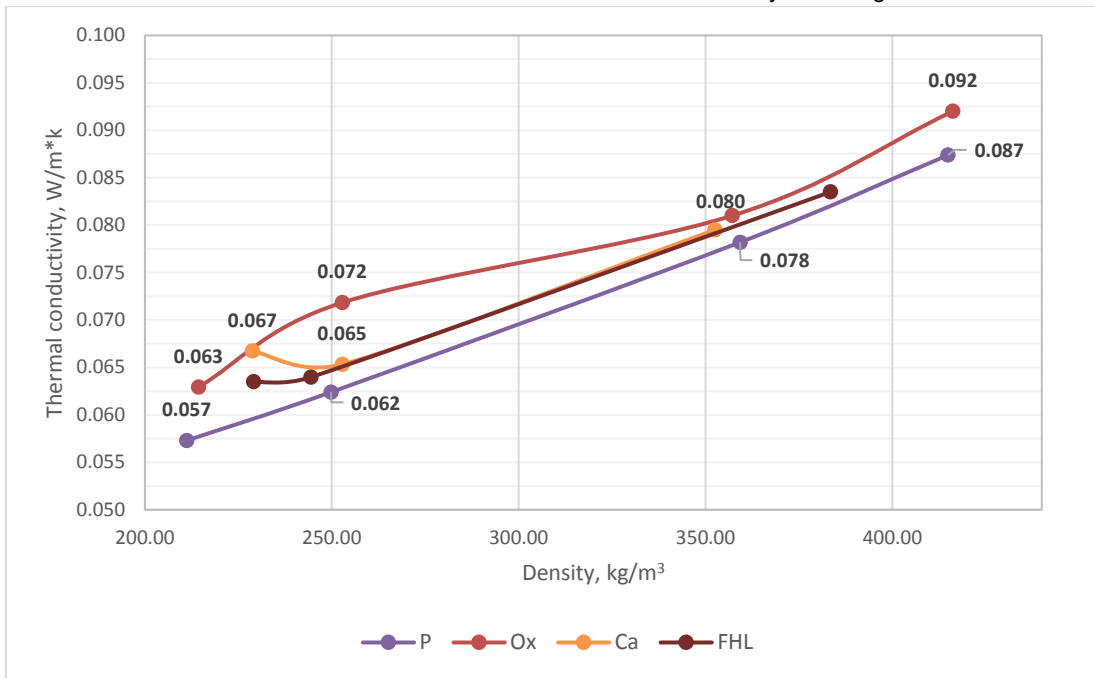

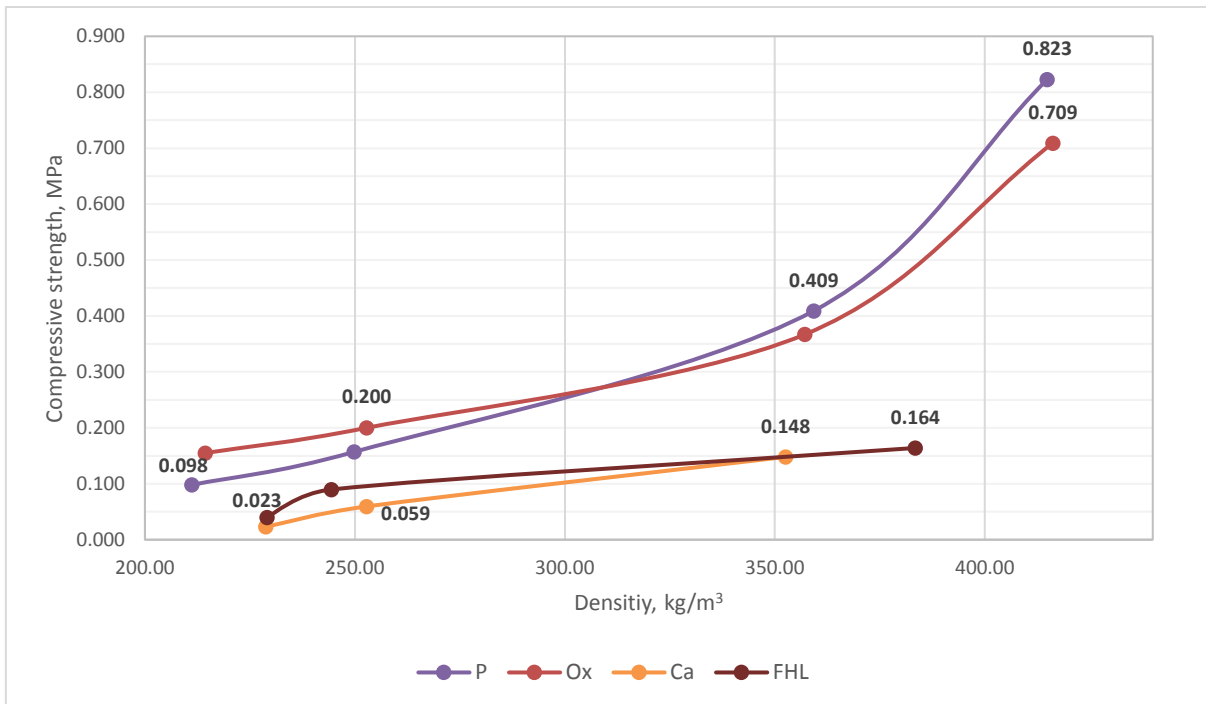*Fig. 5: Thermal conductivity and density of biocomposites with various binders*

Fig. 6: Compressive strength and density of biocomposites with various binders

5 CONCLUSIONS

The following conclusions can be drown from the results:

1. The magnesium orthophosphate hemp composite has the lowest conductivity/density ratio, and it is the highest for the magnesium chloride. Surface area of the dead-burnt magnesia has positive influence on thermal conductivity as there is less of binder compared to composites of similar density. Magnesium oxychloride has higher thermal conductivity than lime based binders because of the fully hardened inner structure of MOC compared to partly unhardened structure of the lime based.

2. MPC and MOC binders have higher compressive strength/density ratio, which could allow their use as self-bearing wall insulation material even at densities of approximately 250 kg/m^2, compared to lime based binders that in turn should have densities around 350 kg/m^3 and higher thermal conductivity.

3. However, lower amount of binder for MPC and MOC composites could cause other limitations,

most notably resistance against bio-degradation as MgO binders have lower amount of pH than lime. MOC is also limited in its use in wet conditions, as the binder is moderately soluble in water and can leach if under constant water action.

4. Addition of magnesium binder to all lime based binders significantly increased their compressive strength due to MgO compatibility with bio based fillers. This addition has only maximum 0,0025 W/m*K impact on thermal conductivity.

5. Adding magnesium binder to the LHC has overall proven to have positive preliminary results, with wide range of possible applications, from self-bearing to low load bearing. However there should also be research of some important properties such as bio resistance and environmental impact of the magnesium binders.

6 ACKNOWLEDGMENTS

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