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THE EFFECT OF GLUCOSE ON THE HYDRATION KINETICS OF ORDINARY PORTLAND CEMENT

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Abstract

In this study, the application of fresh-wood-wool without any pre-treatment to produce wood-wool cement boards (WWCB) is investigated. The aim of this study is to create a binder which is less affected by the inhibitory substances of the wood-wool, without reducing the flexural strength properties of the boards. Moreover, the carbon footprint of WWCB is addressed by not using any waterglass and partly replacing cement with limestone powder in the boards production. First, the hydration behavior of different binders is studied by means of an isothermal calorimeter, including e.g. different types of cement, addition of glucose as a retarder, reduced anhydrite content in OPC and the use of limestone powder as a partial cement replacement. Then, boards are produced with fresh wood-wool and a newly developed binder recipe that is resulted from the hydration behavior analysis. The results show that the retarding effect of glucose is highly dependent on the chemical composition of the cement. In general, high amounts of C_3A and C_3S in a binder are favorable. When available, glucose reacts first with C_3A, hence, less glucose is available to retard the other cement compounds like C_3S. In addition, when using the fresh wood-wool in boards, the strength of the produced boards is increased. Furthermore, the water absorption of the wood is significantly reduced (20%), since it is already physically and chemically bound within the wood-wool. This enables a lower water demand that results in a denser packing of the binder and a reduced ion migration, leading to a mechanical strength increase. Furthermore, without the use of waterglass and the incorporation of limestone powder (36%) as a partial cement replacement, the CO_2 footprint of the developed board fulfills the stated requirements, while possessing a reduced CO_2 footprint.

Keywords:
WWCB; Hydration kinetics, OPC, Glucose, carbon footprint

1 INTRODUCTION

Wood-wool cement board (WWCB), illustrated in Fig. 1, is a wood-cement composite, made from the reinforcing agent wood-wool, Ordinary Portland Cement (OPC) as binder, water as the reactant and small amounts of additives to catalyze the reaction. The wood-wool is coated with cement and pressed into boards that have densities in the range of 400-650 kg/m^3 with properties that fulfill the requirements stated in the EN 13168.

To meet the aesthetic requirements, wood-wool cement board (WWCB) are produced using different cement types varying between white (CEM I 52.5 R white) and grey (CEM I 52.5 R) cement. In case of white cement, the boards can be painted to any desired color based on their aesthetic requirement. Although the chemical composition and created reaction products of these type of binder defer, it is often neglected. Studies performed on wood-cement composites focus more on the inhibitory influence of soluble compounds of the wood-wool on the cement hydration which often is linked to the type of wood (Karade, 2010; Fan et al., 2012; Ashori et al., 2011).

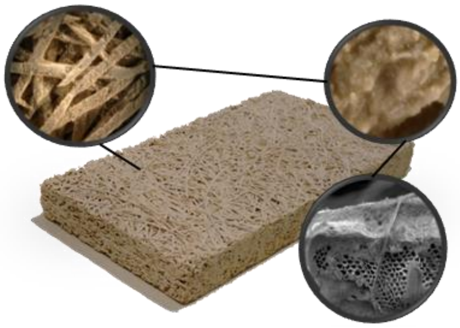

Fig. 1: WWCB created of wood-wool which is mineralized by a white cement creating a protective layer around the wood-wool fiber.

However, as stated by Neville (2002), the content of C_3A which completely reacts with gypsum does not react with a retarder. Hence, the hydration of the calcium silicates is more retarded, which occurs through the adsorption of the retarder onto the calcium nuclei. Furthermore, it is known that C_3A is a compound that binds chloride ions easily. Moreover, Bishop & Barron (2006) indicate that C_3A in combination with sucrose and lignosulfonate accelerate ettringite formation and more retard C_3S hydration while tartaric acid is retarding the C_3A reaction. They indicate that sucrose acts via nucleation poising/surface adsorption while lignosulfonates involve the formation of a semipermeable layer on the cement particles and tartrate by tartaric acid inhibition. In case, C_3A is not retarded but is able to react with the used retarder the following hypothesis is formed: C_3A, which reacts with the used anhydrite to suppress the hydration of C_3A, will in case of a lower content of anhydrite react partly with the retarder (e.g. glucose) and thereby reduce the initial amount of retarder that could react with the calcium nuclei. Hence, the retardation of the other cement compounds is reduced and should be reflected by an increased rate of heat development.

As described by Hachmi & Moslemi (1990), wood-cement compatibility generally decreases with the increase of the wood extractives, which are generally composed of terpenes, fatty acids, tannins, carbohydrates and inorganic materials (Ashori et al., 2011; Poletto et al., 2012). It is known that sugars contained in wood extractives are responsible for the inhibition of cement setting because they prevent the formation of C-S-H gel and the retarding effect is based on the mechanisms of either adsorption or precipitation (Thomas & Birchall 1983). The most common chemical compound in wood is cellulose, which is a crystalline polymer derived from glucose. Glucose itself is a common cement retarder. According to Hewlett (2004), glucose in presence of calcium form calcium glucosate, sealing of the cement particles from water, thus causing retardation.

Currently, three methods are used to overcome the inhibitory effect of wood on cement: (a), by seasoning the wood logs e.g. storing them outdoors for approximately 6 months (Aro, 2004); (b), by water treatment e.g. soaking the wood-wool in a water basin with an increased pH (e.g. by calcium hydroxide or cement) in order to accelerate the dissolution of sugars; (c), by the use of accelerators like chlorides (Fan et al., 2012; Pereira et al., 2006; Hachmi et al., 1990; Simatupang & Geimer, 1990). These accelerators are in practice prohibited due to the WWCB is sometimes applied next to the reinforced concrete where the chloride migration from the boards causes the corrosion of the reinforced concrete.

The aim of this study is to create a binder which is less affected by the inhibitory substances of the wood-wool, without reducing the flexural strength properties of the boards. Moreover, the carbon footprint of WWCB is addressed by not using any waterglass and replacing cement with limestone powder.

Firstly, the heat release of binder is studied with an isothermal calorimeter to evaluate the hydration behavior of different types of OPC and the impact of retarders like glucose. Secondly, the CO_2 footprint of WWCB is minimized by (1) reducing the amount of cement and (2) using no additive like waterglass since it is normally applied to establish a good interfacial transition zone between the wood-wool and binder but has a CO_2 footprint of 403 $kgCO_2$/ton of waterglass (European Commission, 2007). Furthermore, due to the long-time storage of the wood outdoors (which requires large storage space) the applied wood is more brittle, leading to a reduction on both mechanical and aesthetic performance by having more cruelly wood-wool that often is broken to smaller sizes, hence, lowering the bending strength properties of the board. Therefore, boards are produced with fresh wood-wool and the newly developed binder recipe that is resulted from the hydration behavior analysis.

2 MATERIALS AND METHODS

2.1 Materials

Two types of Ordinary Portland Cement (OPC) are studied in this study, which are commercially available under the brand name CEM I 52.5 R and CEM I 52.5 R white (provided by ENCI, NL) (in this study termed *G* and *W,* respectively). The chemical composition of the two cement types determined by X-ray fluorescence (XRF) together with the main cement phases calculated by using the Bogue equations are presented in Tab. 1. *G* has a BET surface area of 1.50 m^2/g and a mean particle size d_{50} of 14.44 μm while *W* is a bit coarser with a BET surface area of 1.21 m^2/g and a mean particle size d_{50} of 16.90 μm. Compared to *G*, *W* has a higher C_3A content (+ 3%) and a higher C_3S content (+ 20%) while with a similar gypsum content (both about 3%). *W* contains almost no iron to ensure the white appearance. Hence, the content of C_4AF is very small (1%). To study the effect of glucose on the hydration of cement, D-glucose (0.2% of mass of cement) is used in isothermal calorimeter measurements. When glucose is used, the variants are indicated with *-g*. Furthermore, the two types of Portland cement are also applied in clinker form (without the addition of anhydrite that normally is used to bond C_3A). These clinker variants are indicated with a *C*, and termed *W_C* and *G_C*. The hypothesis is that the C_3A, which reacts with the used anhydrite to suppress the reaction of C_3A, will in case of a lower content of anhydrite react partly with the glucose and thereby reduce the initial amount of glucose that could react with the calcium nuclei. Hence, the retardation of the other cement compounds is reduced. The used wood-wool in this study has a width of ±1.5 mm, thickness of ±0.03 mm and length of ±25 cm. Both the bulk and specific densities of the wood-wool are determined by using a Micromeritics AutoPore IV 9500, yielding 761 kg/m^3 and 1345 kg/m^3 respectively and having a porosity of 63.85%. The target density of the board is aimed to be 450 kg/m^3. The wood-wool created from wood logs which are stored outdoors for 6 months are termed “old” while the wood-wool which is immediately after delivery of the logs is created and termed “fresh”.

2.2 Measurements

To study the hydration kinetics, tests were performed in a TAM AIR isothermal calorimeter for 90 hours with a water to powder ratio of 0.5 under a temperature of 20 °C. The powder samples were first put in an ampoule and then well mixed before water was added. After the addition of water, the mixture was mixed for 2 min. and the ampoule was loaded to the isothermal calorimeter. The heat evolution rate data were calibrated by subtracting the heat evolution of ampoules with water as a baseline.

Tab. 1: Chemical composition and main Bogue compounds.

Chemical composition	G	W	Compounds	G	W
CaO	63.71	67.19	C_3S	56	80
SiO2	20.27	20.86	C_2S	17	0
Al_2O_3	4.80	3.91	C_3A	7	10
Fe_2O_3	3.43	0.45	C_4AF	10	1
SO_3	2.91	2.92			

In order to investigate if a modified binder would result in an improved WWCB in term of flexural strength, boards were produced with a water to powder ratio by mass of 1.0 and a wood to powder ratio by mass of 0.75. The following lab scale preparation procedure was followed: first, the wood-wool is pre-wetted, to make the wood-wool more flexible and less vulnerable to break. Secondly, the binder is mixed with water to create a homogenous slurry, which is then applied on the wood-wool by means of spraying. Next, the wood-wool cement composite is transferred into a mold and distributed evenly to form the board. Subsequently, the mold is removed, the composite is sealed and compressed to the desired height, which is achieved by using distant holders of 15 mm. After 24 hours compression, the formed boards are stored for 10 days under ambient conditions. Finally, the boards are dried in an oven at 100 °C for 2 hours to remove the capillary water. During the production of the boards it was found that the fresh wood-wool has a higher moisture content (42.6 % compared to 14.69 % for dry wood-wool), hence, the boards were relatively wet compared to wood-wool stored outdoors for 90 days and the initial required water was therefore reduced with 20%.

3 RESULTS

3.1 Hydration kinetics OPC and clinker

Fig. 2 illustrates the heat evolution and heat flow rates of *G*, *W*, *G_C* and *W_C* mixtures, in the first 90 h of reaction. Three distinguishable peaks are observed during the recording period as presented in Fig. 2b. A first sharp peak (within the first 10 min.) appears immediately after mixing and is called the pre-induction period, followed by a second broad exothermic peak which is known as the acceleration period and in case of *W* a sharp third peak which also appears in the acceleration period. The first sharp peak is contributed by the dissolution of particles and fast reaction of C_3A that reacts with Ca^{2+} and SO_4^{2-} ions, yielding the ettringite (Aft) (Taylor, 1990). This product precipitates on the cement particle surface and eventually together with other reaction products slows down the hydration of C_3S and C_2S. C_3A hydrates in the pre-induction period around 5-25% (Taylor, 1990). The reaction of C_3A with the presence of gypsum and water to form ettringite is written as:

$$C_3A + 3C\bar{S}H_2 + (g-6)H \rightarrow C_6A\bar{S}_3H_g \qquad (1)$$

in which: *g* is the amount of water required for the chemical reaction.

When all the gypsum in the paste is consumed by the reaction with C_3A, the ettringite starts to further react with C_3A to form monosulfate as:

$$2C_3A + C_6A\bar{S}_3H_{32} + (3g-32)H \rightarrow 3C_4A\bar{S}H_g$$
(2)

C_3S reacts in the pre-induction period in small quantities, yielding a C-S-H phase and contributes together with C_2S to the Ca^{2+} and OH^- concentration in the liquid phase. The second peak attributes to the reaction of C_3S and the second stage C-S-H starts to be formed. Because of the formation of calcium hydroxide in the liquid phase, the concentration of Ca^{2+} in the liquid gradually declines. The calcium sulphate becomes completely dissolved and the concentration of SO_4^{2-} ions start to decline, due to the formation of the Aft phase. Finally, a third peak appears due to a second stage reaction of C_3A that reacts with ettringite forming monosulphate (Hewlett, 2004). The further increase in heat release of W at 16 h is related tothis product and the following reaction of C_3S.

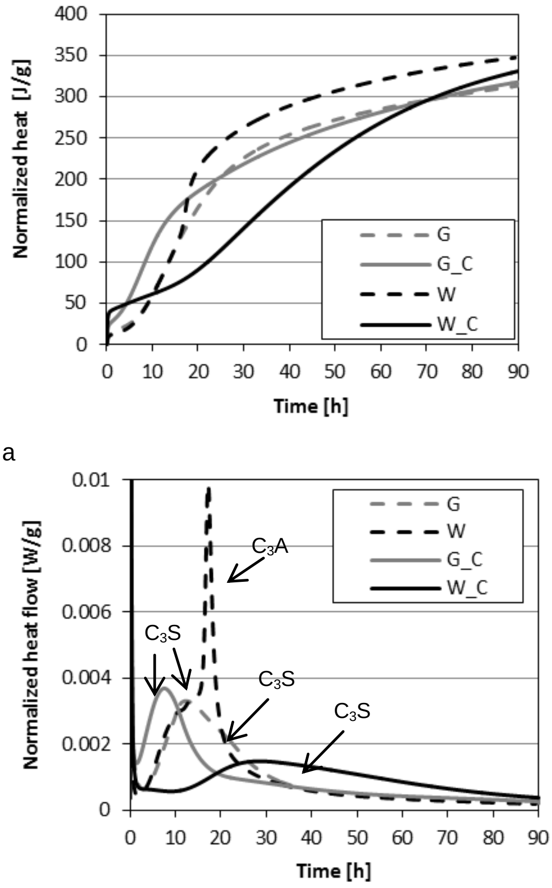

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Fig. 2: (a) normalized heat and (b) normalized heat flow of G, W, G_C and W_C for 90 hours.

In both the clinker variants *G_C* and *W_C* presented in Fig. 2, the heat evolution is in the first 30 minutes 20 and 33 J/g respectively and are significantly higher compared to the variants with anhydrite *G* and *W* (7.5 and 8.0 J/g), due to a higher dissolution of C_3A forming calcium aluminate hydrate (CAH). The deviation between G and W can be related to the higher quantity of C_3A present in *W*. Later, CAH transforms into hexagonal crystals corresponding to the phases C_2AH_8 and C_4AH_8. It is expected that the C_2AH_8 and C_4AH_8 are ultimately converted to a more stable hydrate C_3AH_6. Furthermore, the heat release of *G_C* rapidly increases up to 9 h. On the other hand *W_C* shows, after the pre induction period, a more gradually increase of heat release (18 h) and no second peak can be observed because of the absence of anhydrite, hence, no ettringite formation. The function of anhydrite is to avoid the violent reaction of C_3A, which leads to fast stiffening of the paste. In case of anhydrite is absent (clinker) a flash set (initial high heat release) occurs in which C_3A reacts and precipitates on the cement particle surface retarding the cement hydration (Taylor, 1990). Hence, in case of no anhydrite present, the higher the C_3A content, the more reaction products are precipitated on the cement particles blocking the supply of water and by that reducing the rate of hydration. Therefore, the heat release of *W* is gradually increasing until 18 h, due to the high content of C_3A present. Afterwards the heat release rapidly increases because of the reaction of C_3S.

3.2 Hydration kinetics OPC, clinker and glucose

The heat evaluation and heat flow rate significantly alter when glucose is added to the mixtures, as presented in Fig. 3. The added glucose initially retards both *G* and *W* leading to a very slow increase in heat release until 26 and 43 h respectively and then the heat release starts to increase strongly. The variation in retardation is mainly due to the difference in chemical composition, *G* has a higher content of C_4AF which helps to overcome the retarding effect while *W* has no C_4AF. C_4AF is retarded by the presence of CH and anhydrite forming slowly the phases of AFt that later are transformed to AFm. In case of glucose, this formation continues but C_3S and C_2S are retarded by the glucose and C_3A and C_4AF are retarded by anhydrite forming AFt. Furthermore, C_4AF can react with C_3S forming hydrogarnite. However, the later helps to reduce the duration of the retarding effect of glucose leading to an increased heat release after 26 h. Since, there is no C_4AF present in case of *W* the duration of retardation is longer since C_3A and C_3S are retarded.

In case of *G_C-g* and *W_C-g,* it is clearly demonstrated that C_3A which now is not bound with anhydrite bonds the glucose and increases the early heat evolution of both types to 42 J/g within the first 30 minutes of hydration compared to 24 and 38 J/g for *W_C* and *G_C* and 10 and 11 J/g for *W* and *G* respectively. Afterwards, less glucose is available to bond other cement compounds. However, in the case of *G_C-g*, retardation beyond 90 h is observed due to its lower content of C_3S and missing anhydrite. This is in contrast to *W_C-g* which has a high amount of C_3S that starts to react rapidly after 33 h. Furthermore, it can be observed that due to the fast setting the dissolution and ion-migration at the early age is affected, resulting in a fast set (Collepardi et al., 1984).

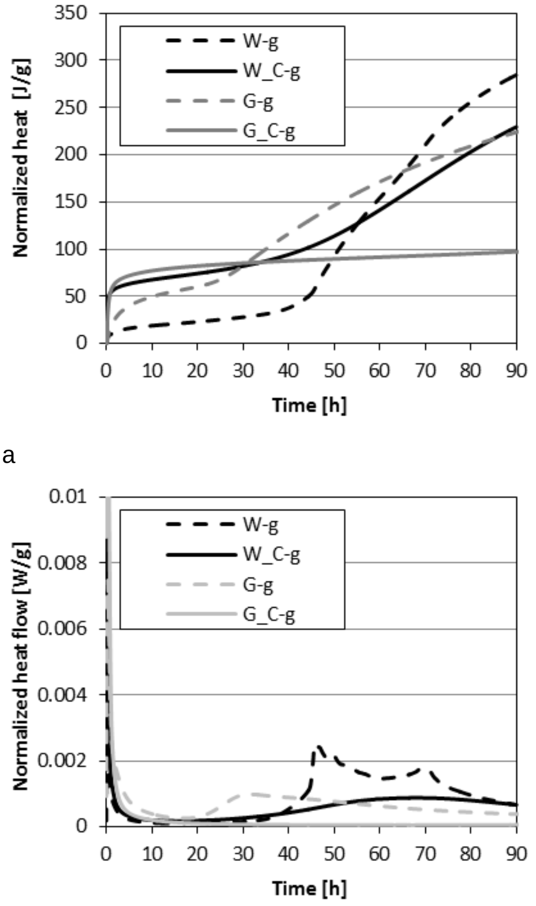

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Fig. 3: Normalized heat and heat flow of G-g, W-g, G_C-g and W_C-g for 90 hours.

3.3 Hydration kinetics OPC, clinker, glucose and limestone powder

In order to reduce the CO_2 footprint of the binder, with the requirement to fulfill the stated requirements in EN 13168, the replacement of white cement (*W*) by 36% limestone powder (L) is studied (*W_L*). The remaining cement is replaced by 20% clinker, termed (*C*) (previously C was representing a 100% clinker) and addition of 0.2% glucose (*-g*) (Fig. 4). Firstly, limestone powder stimulates the reaction of the cement compounds e.g. the heat release of the third peak corresponding to C_3A is increased from 0.01 to 0.21 W/g and acceleration of the hardening can be observed in time (17 h to 13 h) for *W_L*. Furthermore, it can be seen that when 20% clinker is used as cement replacement (*W_L_C*), the reaction is further accelerated (13h to 7.5 h) and the heat release peak of the C_3A is even further increased (0.021 to 0.030 W/g).

When 0.2% glucose is added (*W_L-g*) (based on mass of cement) a retarding effect occurs (3 h less than *W-g*). Furthermore, it is clearly visible that the second C_3A reaction which initially occurs after the C_3S reaction is now shifted and probably changes the structure in terms of porosity of the binder. When 0.2% glucose is added to the 20% clinker variant (*W_L_C-g*) the early reaction is occurring in a high degree and retardation takes place to a less extent. However, it is also observed that the second C_3A peak disappeared. Within one hour of the reaction the peak is visible indicating that the glucose reacted with the C_3A within the first hour of hydration.

Finally, due to the reaction of glucose with C_3A and reaction with limestone powder the reaction takes place before the reaction of C_3S as the second and third heat release peaks of *W_L-g* in Fig. 4 are clearly shifted compared to the second and third heat release peak of *W_L* indicated by the red and blue arrows.

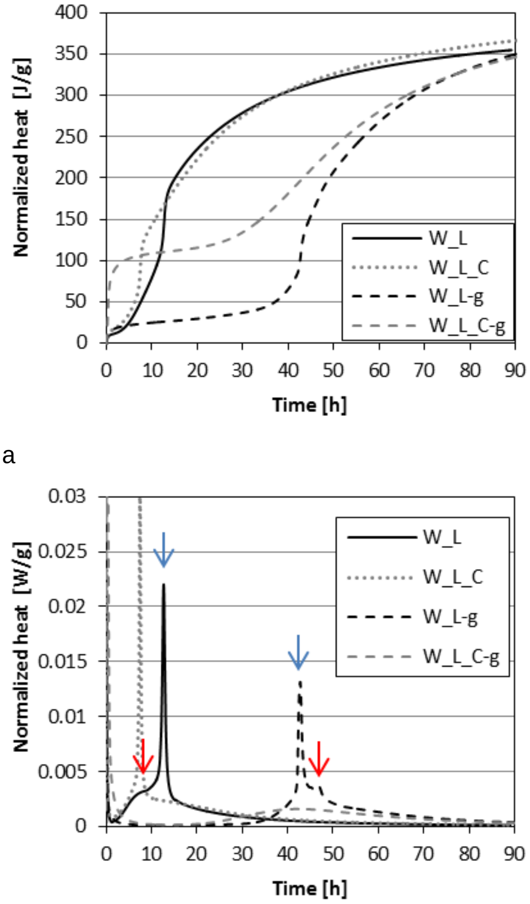

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Fig. 4: Normalized heat and heat flow of white cement (W) replaced by 36% limestone powder (L), 20% clinker (C) and the addition of 0.2% glucose (-g) for 90 hours. The shift of C_3S and C_3A reaction peaks are indicated by blue and red arrows.

From the results it can be concluded that the *W* with high amounts of C_3S and C_3A and 20% less anhydrite can rapidly overcome the influence of glucose and by that it is possible to create boards with sufficient strength within 24 h of hydration.

3.4 Board properties: Flexural strength

Based on the previous results WWCB are produced with the size of 30 x 50 cm^2 (W x L), which is subsequently cut to three boards of 15 x 20 cm^2 (W x L). The flexural strength is tested according to EN 12089. Since the distribution of the boards can lead to varying densities, Tab. 2 only presents boards which have comparable densities and a higher density corresponds with a higher flexural strength. The boards termed 100% W are mixed with fresh wood-wool and show extraordinary strength results compared to commercial available WWCB (termed C_WWCB). This was unexpected since it is believed that the wood-wool has a larger amount of inhibitory substances that retard the cement setting and reduce its mechanical performance. Instead the setting of cement seems to be not affected. A reason for this could be a low content of soluble extractives which is a result of the uncertainty of the age of the shredded wood logs in terms of storage time on the forest site. While from the obtained strength results as well as the observed roughness of the wood-wool it is clear that this wood-wool is “more” fresh compared to the old wood-wool, of whichthe level of freshness is unknown. This later is confirmed by the chemical analysis presented in Tab. 3 of the wood-wool which show very comparable chemical compositions. The boards in which fresh wood-wool and clinker was used (termed 80% W+ 20% C (wet)) show strength results exceeding all other boards indicating the positive influence of the clinker by increasing the setting of cement while keeping a similar heat of reaction. The use of old wood-wool presented lower mechanical strength properties compared to fresh wood-wool. However, when the boards are put in an oven for 24 h at 100 °C, the difference in flexural strength is found to be negligible. Finally, the boards produced with fresh wood-wool, with 36% limestone powder and 20% less water have an oven dry density of 450 kg/m^3 and a flexural strength >2 MPa,fulfilling the requirements stated in the EN 13168. The reduced water amount is based on the high moisture content present in the fresh wood-wool and the reduced cement amount, since limestone powder has a lower water absorption (18% per gram of cement) compared to cement.

Tab. 2: Produced WWCB with their density and flexural strength properties.

Boards	Density [kg/m^3]	Flexural strength [MPa]
100% W (wet)	520.7	5.9
100% W (wet)	550.6	6.5
C_WWCB	455.7	3.0
C_WWCB	538.5	3.9
C_WWCB	570.5	4.9
80% W+ 20% C (wet)	443.3	3.6
80% W+ 20% C (wet)	528.8	7.1
64% W+ 36% L (wet)	399.9	1.9
64% W+ 36% L (wet)	392.9	1.8
*80% W+ 20% C (wet)	423.8	1.9
*80% W+ 20% C (wet)	451.6	2.9

** Dried samples with a 20% lower moisture content*

Tab. 3: Chemical composition of the used Spruce wood-wool.

Constituent	Fresh [%]	Old [%]
Hemicellulose	21.6	24.4
Cellulose	41.0	38.7
Lignin	33.5	32.7
Extractives	4.0	4.7

4 CONCLUSSION

This paper addresses the hydration kinetics of cement with glucose as a strong retarder to research the binder composition that can overcome the retarding effect. Furthermore, the newly designed composite is tested by applying fresh wood-wool to produce WWCB. Based on the obtained results, the following conclusions can be drawn:

- The function of anhydrite is to suppress the reaction of C_3A, leading to a more gradually reaction, resulting into an higher heat release after 90 h compared to a flash set when less anhydrite is used.
- The inhibitory effect of glucose on cement is depending on the chemical composition of the used cement. In case of OPC (with anhydrite), both C_3A and C_4AF form ettringite and will not affect the hydration of the calcium silicate phases and will in a low degree retarded by glucose. In case of OPC which does not contain C_4AF and instead has a higher C_3S content will be strongly retarded by glucose, although in a later stage a rapid heat is developed.
- In case clinker (no anhydrite present) coverage of the cement phases C_3S and C_2S is observed due to the reaction of C_3A and C_4AF reducing the rate of hydration because reaction products precipitate on cement compounds blocking the supply of water and stiffening the paste. The rate of blocking depends on the amount of C_3A and C_4AF present, the higher the content the longer the duration of this phenomena will occur.
- In combination with glucose and clinker. C_3A bonds glucose, increasing the heat release and reducing the retarding effect. In case less C3A is present, a higher percentage of glucose can react with the remaining phases increasing the retardation. Furthermore, in case of OPC with C_4AF the duration of the retarding effect increases due to fast reaction of C_4AF.
- In combination with limestone powder, the reaction of white cement is accelerated.
- The higher the C_3S amount, the faster the heat release after the retardation effect.

Regarding the produced boards and their flexural strength, it can be concluded that:

- The fresh wood-wool which initially was used to evaluate the retarding effect on the binders does not show a significant impact on the binder.
- Fresh wood-wool compared to old wood-wool show a higher roughness, making it less brittle which can lead to a higher flexural strength.
- The produced boards show comparable or higher strength results compared to the commercially available WWCB.
- The use of limestone powder as a partial cement replacer to produce boards with a density range of 450 kg/m^3 and a strength >2 MPa fulfilling the requirements stated in the EN 13168.

The synthetic conclusion regarding the main studied points is that incorporation of limestone powder and reducing the anhydrite amount lead to accelerated reactions and by that the retardation influence of glucose is able to be reduced. However, the reduction of anhydrite and amount of limestone powder needs to be tailored in order not to result in a binder which shows a very fast reaction, in addition the amount of glucose applied is also strongly revelant.

5 FURTHER RESEARCH

The further research will be conducted on the initial moisture and extractive content of wood logs in order to validate the performance of replacing cement with 20% clinker and the use of 36% limestone powder without the use of additives. Furthermore, a study will be performed to select wood logs based on their moisture content, to investigate the relation betweenthe mechanical performance of the wood-wool and the initial moisture content of the wood logs.

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