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The Influence of Wood Extractives and Additives on the Hydration Kinetics of Cement Paste and Cement-bonded Particleboard

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Abstract: The experiment was carried out to study the effect of wood extractives, chamotte and $CaCl_2$ on hydration and hardening of cement paste. It also allowed study of the effects of these additives on mechanical and physical properties of Cement-bonded Particle Board (CBPB). Homogeneous cement-bonded particleboards 12 mm in thickness were made from poplar flake with Portland cement, calcium chloride and chamotte. The CBPB was manufactured with; a wood/cement (w/c) ratio of 0.3:1 giving a specific gravity of 1.2 kg m^{-3}; $CaCl_2$ content as cement replacement of 3, 5 and 7%; chamotte content as cement replacement of 5, 10 and 15%. The CBPB was tested for Modulus Of Rupture (MOR), Internal Bond (IB), Thickness Swelling (TS) and Water Absorption (WA). The results showed that water-soluble wood extractive increases the hydration time of cement. It was observed that different amounts of chamotte and $CaCl_2$ in neat cement can significantly affect the setting and hardening time. Replacement of cement with 10% of $CaCl_2$ and 10% chamotte in boards increased the MOR and IB. It also decreased WA and TS but most importantly eliminated the inhibitory effect of wood on cement setting and hardening.

Key words: Cement-bonded particle board, alder, poplar, calcium chloride, chamotte, hydration

INTRODUCTION

Wood is composed of cellulose, hemicelluloses, lignin, extractives and soluble sugars. Soluble sugars and part of hemicelluloses which under certain conditions can be resolved, have a negative effect on cement's hydration (Vaickelionis and Vaickelioniene, 2003; Frybort *et al*., 2008). Therefore, the wood to cement compatibility is still a problem to Cement-Bonded Particle Board (CBPB) development. The term compatibility, when applied in the research area of wood cement composites, refers to the degree of cement setting after mixing with water and with a given wood in a fragmented form Jorge *et al*. (2004).

The presence of inhibitory substances such as starches, sugars, hemicelluloses and extractives in wood are known to affect and influence cement curing/setting time (Wei *et al*., 1999). Studies have shown that the degraded polysaccharides become a major factor and played an important role in inhibiting the setting of cement (Wei *et al*., 2002). A similar effect on delay in the hydration of cement can be observed when composites are made using wood.

While extractives in wood prolong the setting time of cement (Singh and Garg, 1997) adding an accelerator into a mixture of cement-water could decrease cement's setting time. Addition of an accelerator into a mixture of cement-water paste could enhance the speed of hydration process to occur earlier. This was confirmed by previous studies (Xu and Stark, 2005) where they add accelerator into a mixture of OPC-water and found out that the accelerator caused early hydration. According to Maltese *et al*. (2007), the addition of $CaCl_2$ and $MgCl_2$ increased the temperature of the mixtures and encouraged the activation of cement hydration which later gives a more dense paste structure with smaller pores compared with the Portland cement neat. Therefore, as the concentration of $CaCl_2$ and $MgCl_2$ are increased, the activation becomes more rapid and dense paste structure could be achieved earlier (Azrieda *et al*., 2009).

The positive side effect of using accelerators is the fact that the possibility of washing out aggressive extractives from the wooden particles is minimized and therefore mechanical properties are increased as well (Frybort *et al*., 2008). Various additives to the cement have been reported to shorten the setting time of wood-cement mixtures (Moslemi *et al*., 1983). One of the materials that can be used as an additive in CBPB is chamotte. Because of the abundant supply of chamotte and its comparative cheapness, it can be extensively used to produce CBPB.

Wood can not combine well with cement unless it has been pretreated. While the precise mechanism of

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cement setting in the presence of wood particles is yet to be fully understood, several means of minimizing the effect have been devised, including prolonged storage of the wood material, hot or cold water extraction of soluble sugars and the use of chemical accelerators, namely dilute sodium hydroxide (NaOH), sodium silicate (Na_2SiO_3), calcium chloride ($CaCl_2$) and aluminum sulphate ($Al_2(SO_4)_3$), among others (Olorunnisola and Adefisan, 2002). It seems that the use of $CaCl_2$ and chamotte tend to accelerate the hydration of wood-cement mixtures, thereby enhancing the wood-cement bond and the mechanical properties of the composites. Therefore, the purpose of this present study is to study the influence of the wood extractives and accelerators on the setting and hardening properties of cement and the influence of the $CaCl_2$ and chamotte on the mechanical and physical properties of CBPB.

MATERIALS AND METHODS

This study was carried out at the Department of Wood and Paper Science and Technology, University of Zabol (Iran), from beginning of the autumn 2009 to end of the autumn 2010. Poplar and alder particles that previously passed the 1×1 mm sieve, as the raw material and additives ($CaCl_2$ and chamotte) in three levels were used in this study. The cement used was type I Portland. Prior to board fabrication, all particles were soaked in hot water (50°C) for 2 h to reduce the amount of water-soluble sugars and also to investigate the effect of water soluble extractives on cement's hydration. After that the particles were finally dried to approximately 3% moisture content.

At first, the effect of alder and poplar extractives on the setting time and compressive strength of hardened cement was determined by EN 196-3 (2005). For this aim, Water-soluble alder and poplar wood extractive were mixed with cement. According to EN 196-3 (2005), the initial and final setting times of neat cement paste containing water-soluble wood extractives were determined by Vicat apparatus. After that, compressive strength at 28 days was determined by using 20 mm cubes in size.

In order to understand the affect of mineral additives on physical and mechanical properties of CBPB made from alder and poplar particles, chamotte was considered for this study due to presence of minerals such as alumina, calcium and magnesium oxide. These minerals make chamotte an ideal aggregate for faster setting of cementitious materials. Specimens of chamotte-rich cement were made using Portland cement and chamotte of size ranging from 10 to 500 μm in diameter. Some weight fractions of chamotte (0-20%, in increments of 5%) were considered in this study. For all the cement specimens, the water to cement ratio was maintained at 0.3. Vicat apparatus using neat cement paste containing chamotte determined setting and hardening times.

In this study, poplar and alder veneers, treated with hot water (2 h at 50°C), were employed. Air-dried and weighed veneers (300×70×0.4 mm) were each placed in a container of cement paste with a different level of mixed chamotte and distilled water. The percentage of absorbed paste on these veneers was then determined after 24 h of soaking. After that, the samples were conditioned at 20±2°C and 65±3% relative humidity until their weights became stable by storing them for two weeks in the conditioning room. After this time, test samples had 12% average moisture content. The samples were weighed and the percentage of chamotte on the veneers was determined by using the following equation:

$$A\ (\%) = \frac{(W_2 - W_1)\times 100}{W_1}$$

where, A is the chamotte absorption content, W_1 is the weight of the conditioned veneer before impregnation and W_2 is the weight of veneer after impregnation and conditioning.

In order to manufacture the CBPB, poplar wood particles were mixed with cement (particles to cement ratio of 1:3) by weight with additives in different levels (Table 1). The wood particle was wetted with water plus chloride calcium solution and then cement-chamotte admixture was added. After 15 min of manual mixing, the cement-wood water mixture was screened onto to a caul. Nine types of panels were produced under the laboratory conditions. A total of 27 mats with dimensions of 400×400 mm were manually formed in a frame prior to pressing. Cold pressing took place under an initial pressure of 15 MPa, to a 20 mm thickness, after which the board was retained in compression for 48 h. Target board density was 1200 kg m^{-3}. To minimize cement capillary desiccation and enhance hydration, boards were misted with distilled water, then wrapped in cellophane before storing for curing at 20°C and 60% RH for a month. They were then tested for MOR (modulus of rupture), IB (internal bonding), TS (thickness swelling), WA

Table 1: The content of portland cement, chamotte, chloride calcium and wood particle used in 1 m^3 CBPB

Components	Amount of material
Wood particle (kg)	300
Cement (kg)	597-700
Ch/C ratio (%)	0-20
$CaCl_2$/C ratio (%)	3-7
Water (L)	400

(water absorption, after 24 h immersion in water) according to procedures defined in the European Union Standards, EN 319 (1993), EN 317 (1993) and TS-EN 310 (1999).

Data for each test were statistically analyzed. The second order plan was used to test the significant difference between factors and levels. When the second order plan indicated a significant difference among factors and levels, a comparison of the means was done employing a regression equation to identify.

RESULTS AND DISCUSSION

Setting and hardening experiments of cement paste: The results of the hydration test suggest that the mechanical properties of boards manufactured from certain raw materials and additives can be predicted with a reasonable degree of accuracy using the degree of hydration of the cement-bonded composites. Therefore, at the first stage of the experiment, the effect of alder and poplar wood extractives was investigated. The results obtained from the hydration test showed that water-soluble wood extractive increases the hydration time of cement. The percentage of wood extractive in the alder probably is higher than poplar. Therefore, Fig. 1 shows that cement mixed with the alder's extractive had higher hydration time (430 min) compared to the cement mixed with poplar's extractive (380 min). It was also determined that extractives in cement paste decreased the compression strength of cement stone with respect to control samples (Fig. 2). Hardened cement containing poplar's extractive showed higher compressive strength (53 MPa) in comparing to cement mixed with poplar's extractive (46 MPa). It may be due to existence of lower extractives in poplar wood. Similar results have been observed in several previous studies that water soluble wood's extractive slows down hardening and hydration of cement. A study by Vaickelionis and Vaickelioniene (2003) showed that the amount of chemically combined water (water that participates in the reaction) determines the degree of cement's hydration. They concluded that extract's additives in cement decrease the amount of chemically combined water in cement stone, while the decrease of amount of chemically combined water is not big, when cement is hardened in natural conditions. Additionally, in the presence of extractives, impermeable hydrates are formed around unhydrated cement grains which inhibit or delay the setting of cement. With increasing extractive content, the proportion of unhydrated cement clinker also increases which leads to strength reduction of the cement-wood composite (Wei *et al*., 2003). The investigation has confirmed that water-soluble wood sugars slow down the hardening and hydration of cement in CBPB and this harmful influence can be decreased using mineral additives (for example, chamotte).

Figure 3 shows the effect of mineral additives on the setting time of cement paste. It was observed that the amounts of chamotte in neat cement affect setting time. Increments in the chamotte/cement ratio resulted in a decrease in setting time. Also, the effect of this additive on the hardening time of cement paste is shown in Fig. 4. Hardening time was decreased with increasing the amounts of chamotte in cement paste. In addition, higher proportions of chamotte in the cement paste enhanced its setting and hardening property (20% weight on cement).

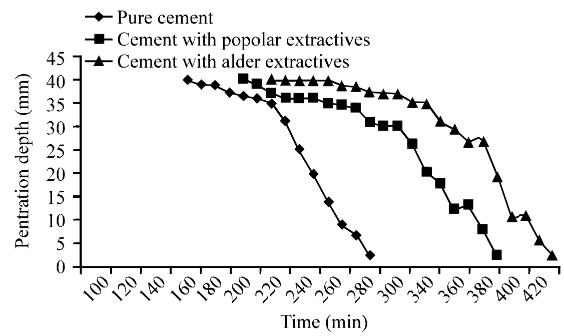

Fig. 1: Penetration depth in mm (measured with the same water/binder ratio)

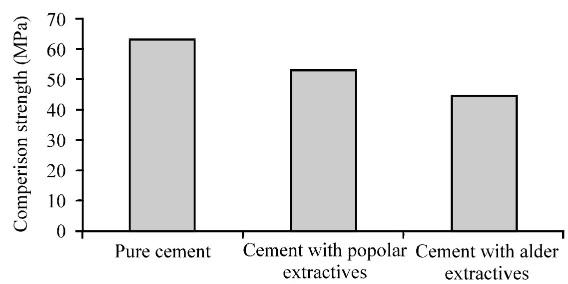

Fig. 2: The effect of alder and poplar wood extractives on compression strength values of cement stones with respect to control samples

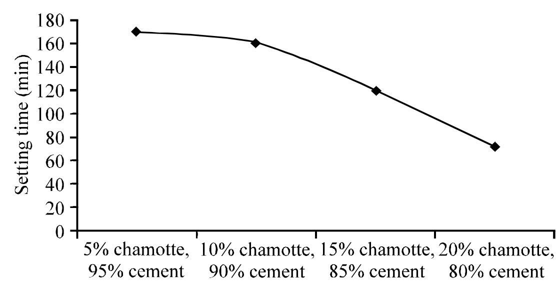

Fig. 3: Setting time of cement paste containing different amounts of chamotte

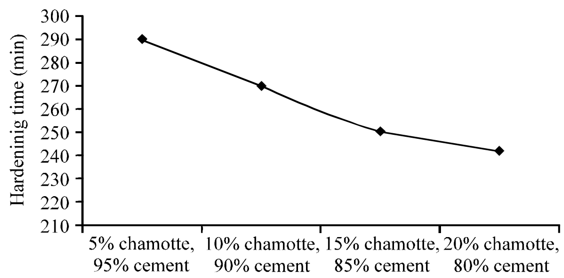

Fig. 4: Hardening time of cement paste containing different amounts of chamotte

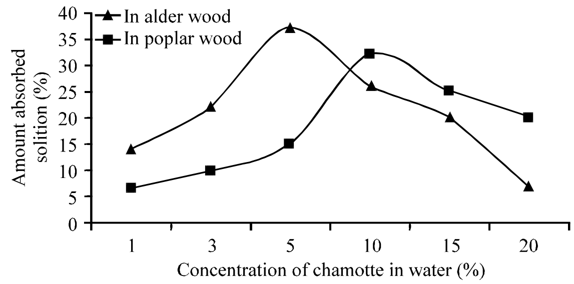

Fig. 5: Concentration-dependent adsorption of chamotte onto alder and poplar wood veneers after immersion in chamotte paste

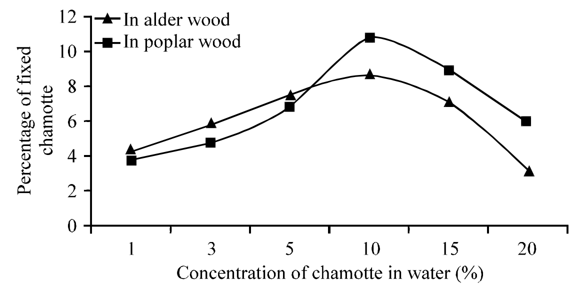

Fig. 6: Concentration-dependent adsorption of chamotte onto alder and poplar wood veneers after immersion in chamotte paste and dried for two weeks

It has hypothesized that the properties of the water film which separates the cement grains in a cement paste, differ from those of the water film that separates the cement and chamotte grains in a cement-chamotte paste. The cement grains are coated with a layer of gel-like hydrates, whereas the chamotte grains are coated with a layer of adsorbed water of high density 1.2-2.0 g cm^{-3} (Akhverdov, 1981). As is known, the compaction and shrinkage of gel structures occur at a higher rate in viscous pastes containing a smaller amount of water (Lea, 1961). Therefore, the shorter setting time for the cement-chamotte paste is due to the decreased amount of free water cased by the buildup of adsorption layers of water on chamotte grains. In this case, similar results have been observed in previous studies. Goberis and Pundene (2005) studied hydration of Gorkal-70 cement in cement-chamotte pastes and showed that because of the buildup of adsorption layers of water on chamotte grains, the paste becomes more viscous. Vaickelionis and Vaickelioniene (2003) studied the influence of organic and mineral additives on hydration of cement and determined that mineral additives eliminate harmful influence of extractives in wood on the cement setting.

Concentration-dependent adsorption of chamotte onto alder and poplar veneers are shown in Fig. 5 which indicate an increase in the chamotte uptake as the concentration of chamotte increases from 0 to 10%. However, the higher concentration of chamotte (from 10 to 20%) decreases the adsorption of chamotte onto veneers. At 5% chamotte, alder wood veneer absorbed a higher amount of chamotte paste (36%) than the poplar wood veneer (15%). But at 10% chamotte concentration, poplar wood veneer absorbed a higher amount of chamotte paste (32%) than the poplar wood veneer (26%). In contrast, after conditioning treatment at 20°C and 65% relative humidity for two weeks, alder wood veneer absorbed lower chamotte than poplar wood veneer (Fig. 6). In this condition, alder and poplar veneers treated with a paste containing 10% chamotte had the most absorptions of 8.5 and 10.5%, respectively.

The optimum range for the water/cement ratio is dependent, in part, on the cement/chamotte ratio. Because the chamotte grains are coated with a layer of adsorbed water of high density 1.2-2.0 g cm^{-3}, as the water/chamotte ratio increases from 80:20 to 95:5, the concentration of chamotte paste decreases. At the lower water/chamotte ratio (80:20), the available water is insufficient for a good penetration of chamotte paste into veneers. On the other hand, at the higher water/chamotte ratio (95:5), too much water is present, the concentration of paste decreases and the paste cannot be well absorbed (or fixed) in veneers. Therefore, the optimum range for absorbance by wood veneers is a cement/chamotte ratio of 90:10. When $CaCl_2$ was incorporated, the mixture was graded as low inhibition. The effect of value of inhibitory index is negative, probably due to the capacity of the $CaCl_2$ to buffer or minimize the adverse effect of the soluble sugars, extractives and also to accelerate the cement hardening and setting (Papadopoulos, 2008). From this it can be seen that the mixture of $CaCl_2$-chamotte-cement was classified as having moderate inhibition.

Mechanical and physical properties: Table 2 shows the mean MOR values of the composites treated by 5-15%

Table 2: Physical and mechanical properties of cement-bonded particleboard

Additives% (g)					
$CaCl_2$	Chamotte	MOR (MPa)	IB (MPa)	TS (%)	WA (%)
3 (19.9)	5 (35)	11.16*	0.32*	1.98	18.9
7 (46.5)	5 (35)	10.41*	0.34*	2.80*	20.3
3 (17.8)	15 (105)	10.80*	0.29*	3.06*	22.9
7 (41.6)	15 (105)	9.20	0.32*	3.95*	25.8
3 (18.9)	10 (70)	12.96*	0.33*	2.53*	20.2
7 (44.1)	10 (70)	11.71*	0.36	3.43*	22.0
5 (33.2)	5 (35)	13.70*	0.39	1.56	14.2
5 (29.7)	15 (105)	12.80*	0.36	3.40*	17.2
5 (31.5)	10 (70)	14.90*	0.40	1.73	15.5
EN 310, EN 317 and EN 319		9.00	0.40	1.2-1.8	---

*Mean difference is significant at the 0.05 level compared with EN standard

chamotte and 3-7% $CaCl_2$. Composites produced by 70 g chamotte and 31 g $CaCl_2$ had the highest MOR, followed by those manufactured with 5% (35 g) chamotte and 5% (33.2 g) $CaCl_2$. A strong negative correlation (Y_1 = -9.167-0.52 x_1+0.403 x_2+2.833 x_1^2+1.945 x_2^2-0.2125 x_1x_2) between content of $CaCl_2$ (x_1) and chamotte (x_2) in boards showed that chamotte and $CaCl_2$ had significant effect ($p<0.05$) on the MOR of the composites. This indicates that the addition of $CaCl_2$ is more effective than chamotte in order to improve the MOR of the wood-cement composites because the coefficient of x_2 is higher than x_1. Additionally, all boards containing 3, 5 and 7% $CaCl_2$ and 5, 10 and 15% chamotte met BISON type HZ requirements for MOR which specifies 9 MPa.

Among the chemical additives, chlorides such as $CaCl_2$ and $FeCl_3$ markedly accelerate the hydration process when they add to cement paste (Wei *et al*., 2000). Some studies reported that the additives such as chlorides could be used effectively as accelerators to restrain the inhibitory influence of wood species (Moslemi *et al*., 1983; Sauvat *et al*., 1999). Wei *et al*. (2000) showed that $CaCl_2$ is an acceptable accelerator because it produces maximum hydration temperatures of above 50°C with the addition of 4%. Chen and Hwang (1998) investigated effect of hot water extraction wand the addition of $CaCl_2$ on the inhibitory characteristics of five hardwood species in CBPB and showed that both hot water extraction and the addition of calcium chloride can improve the compatibilities of five hardwood species with Portland cement, especially the addition of calcium chloride substantially reduces more than half the setting time of highly inhibitory species. Also, the mechanical properties of CBPB with 3% calcium chloride addition were the best. Olorunnisola (2007) had investigated the effects of $CaCl_2$ on rattan-cement composites and reported that the $CaCl_2$ treated samples general had higher MOR than the untreated samples.

As shown in Table 2, the IB value was significantly decreased, as the content of chamotte and $CaCl_2$ increased to more than 10 and 7%, respectively. Also, when the content of chamotte and $CaCl_2$ decreased to 5 and 3%, respectively, the IB value was significantly decreased. For specimens made with 10% chamotte and 5% $CaCl_2$, the IB has maximum values (about 0.40 MPa). A negative correlation (Y_2=-0.401-0.013x_1+0.013x_2+0.156x_1^2+0.096x_2^2-0.002x_1x_2) between content of $CaCl_2$ (x_1) and chamotte (x_2) in boards showed that chamotte and $CaCl_2$ had significant effect ($p<0.05$) on the IB of the composites. Additionally, only boards containing 5% $CaCl_2$ and 10% chamotte met BISON type HZ requirements for IB which specifies 0.4 MPa.

Ahn and Moslemi (1980) observed conical-shaped crystals of hydrated cement embedded in wood and concluded that their interlocking improved the bond between wood and cement and hence improvement in strength. The additive of mineral materials (e.g., chamotte) to the cement mixtures decreases the influence of wood's extractives. Because the specific surface of these mineral additives (400 m^2 kg^{-1}) is much bigger than the cement's (300 m^2 kg^{-1}) and the sorption power of these materials is increased. The adsorption of water-soluble wood material first takes place on the surface of additives and extract concentrations in cement paste decreases. Therefore, water easily gets to the grains of cement and the hydration conditions become more favourable, producing a positive effect in the form of an increase of IB and MOR. There is a favorable identity between hydration characteristics and MOR and IB values. Because of existence of mineral components such as $CaO.SiO_2$ and Fe_2O_3 in chamotte, the strengths contributed by C_3S ($3CaO.SiO_2$, tricalcium silicate) and C_2S ($2CaO.SiO_2$, dicalcium silicate) increase under a high hydration temperature (Bogue, 1964). In this case, Ramachandran (1994) reported that $CaCl_2$ achieves improve the strength properties of CBPB by accelerating the hydration of cement particularly the tri-calcium silicate phase (C_3S), reducing setting time and in some cases, increasing maximum hydration temperature. Del Menezzi *et al*. (2007) observed an improvement of mechanical properties as well as mitigation of thickness swelling when fumed silica

(10%) was added. According to the results obtained by Ahn and Moslemi (1980), the bending strength of CBPB was nearly quadrupled compared to controls when 3% $CaCl_2$ was added, because the possibility to wash out poisonous extractives from the wooden particles is minimized (Frybort *et al.*, 2008).

The lowest TS and WA values were obtained from the board with 5% (33.2 g) $CaCl_2$ and 5% (35 g) chamotte, followed by those manufactured with 5% (31.5 g) $CaCl_2$ and 10% (70 g) chamotte (Table 2). Boards at this level are the most dimensionally stable. It is observed that the TS and WA of boards were affected by the production variables used in board manufacture. However, the TS and WA of boards increased as $CaCl_2$ and chamotte increased to 7 and 15%, respectively. It was also observed that the TS and WA of boards increased as $CaCl_2$ decreased from 5 to 3%.

The results of tests showed that increase of $CaCl_2$ and chamotte from 3 to 7% and 5 to 15%, respectively, have a significant effect on the physical properties of boards. At the various interactions, there are also significant differences in the TS and WA of the boards. Positive correlations (Y_3= 2.173+0.482 x_1+ 0.481 x_2-0.874 x_1^2-0.193 x_2^2+0.116x_1x_2 and Y_4 = 8.403+1.016 x_1+1.916 x_2+2.69 x_1^2+ 1.34 x_2^2+1.368 x_1x_2) between content of $CaCl_2$ (x_1) and chamotte (x_2) in boards showed that chamotte and $CaCl_2$ have a significant effect ($p<0.05$) on the water absorption and thickness swelling of the composites. Additionally, only boards containing 5% $CaCl_2$ and 5-10% chamotte met BISON type HZ requirements for TS which specifies 1.2-1.8%.

Del Menezzi *et al.* (2007) found that a negative correlation for MOR and parallel compression concerning TS. This means that higher MOR values are associated with lower TS. Also internal bonding is correlated with TS. Panels with high IB can resist the stress due to wood expansion which results in lower values for TS. The chemical additives can decrease TS. Similar results have been observed in previous studies that chemical additives cause decrease in TS of the wood-cement composite material. By using $CaCl_2$ as an accelerator, TS could be reduced. This improvement is explained by better fiber to fiber contact as a result of improved bonding ability with cement (Frybort *et al.*, 2008). Besides $CaCl_2$ also hot water soaking or $MgCl_2$ treatment are effective methods to reduce TS (Semple and Evans, 2004).

CONCLUSION

Wood extractives retard the hydration of Portland cement. It was determined that alder and poplar wood extractives increase the hydration time of cement paste and decrease the amount of compression strength of cement stone with respect to control samples.

The hydration behaviors of neat cement samples were found to be different and strongly influenced by quantities of chamotte. Twenty percent of chamotte based on the cement weight was the best level of additive to improve hydration properties.

The optimum chamotte content based on the cement weight for the adsorption of wood veneer was between 5 to 10%. Increment (more than 10%) and decrement (lower than 5%) in chamotte-cement ratio resulted in decrease in setting and hardening time.

Composites produced by 10% chamotte and 5% $CaCl_2$ had the highest MOR, followed by those manufactured with 5% chamotte and 5% $CaCl_2$. It is indicated that the addition of $CaCl_2$ is more effective than chamotte in order to improve MOR of the wood-cement composites. The IB value was significantly decreased, as the content of chamotte and $CaCl_2$ increased to more than 10 and 7%, respectively. Also, when the content of chamotte and $CaCl_2$ decreased to 5 and 3%, respectively, the IB value was significantly decreased. For specimens made with 10% chamotte and 5% $CaCl_2$, the IB had maximum values (about 0.40 MPa).

It is observed that the TS and WA of boards were affected by the production variables used in board manufacture. The lowest TS and WA values were obtained from the board with 5% $CaCl_2$ and 5% chamotte, followed by those manufactured with 5% $CaCl_2$ and 10% chamotte.

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