Softwood surface compatibility with inorganic geopolymer

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Abstract

Research describes softwood – pine (Pinus sylvestris L.) and spruce (Picea abies [L.] Karst) and hardwood – birch (Betula pendula Roth) binding with inorganic geopolymer binder, the main focus is on the softwood and geopolymer binding principles. Geopolymer binder is formed from calcined clay and liquid glass. The research describes geopolymer binder compositions with various ratios of liquid glass and clay. This research focuses on mechanical strength of bonded wood samples in shear test and on visualization of binder in three dimensions using X-ray computed tomography with submicron resolution. The scan results are compared with mechanical strength test results. The results of the research shows, that inorganic binder with spruce wood can achieve 7 MPa shear strength. The research describes binding principles that function between wood and geopolymer and explains the main reasons for destruction of joint.

Keywords: Geopolymer, wood, sodium silicate, clay.

Introduction

Along with the development of ecological and environmentally friendly architecture the wood has become a renewable element in concept of green building. One of the disadvantages of wood is its low fire resistance, which requires searching for effective solutions to prevent it. At the same time, geopolymer is an alternative material of concrete with lower CO_2 emissions [1] and higher thermal resistance. This paper looks at wood-geopolymer composite as one of the possible solutions to combine these two environmentally friendly materials. The present research is intended to design wood or wood material that is coated with a geopolymer or wood-geopolymer coating as non-combustible or hardly combustible material that protects the wood from burning. Previous studies have shown good reaction to fire of wood particles and geopolymer composite could be recommended for constructions protection against fire in out-doors and indoors [2].

Geopolymers are amorphous three-dimensional alkali alumosilicate materials that can be used as an eco-friendly alternative to cement. They contain aluminium (Al) and Silicon (Si) compositions, which are soluble in strong alkaline solutions. Any material rich with Al and Si in amorphous form may serve as a raw material of geopolymer [3-5]. Previous studies have focused mainly on the mechanical characteristics of geopolymers and their use as an alternative to concrete [5]. Formation of geopolymer and the characteristics of the final product are affected by various factors: thermal treatment of raw materials [6-8], alkaline activator [3], water [9-11], particle size [11], temperature [12], hardening time [6], and composition (concentrations) [9; 10; 13]. Due to the low temperatures required for obtaining of geopolymers, they can be combined with wood or lignocellulose in a single composite material.

Existing studies have paid a little attention to the wood and geopolymers composites. This study is a part of a new wood and geopolymer composite development [2, 14]. To improve the composite properties, it is essential to ensure connection between separate components – between the wood and the geoplymer. In this work, binding of pine and spruce wood with geopolymer made from calcined clay and sodium silicate solution is examined.

Currently, studies on geopolymers are dominated by a fine ash and various slag, but Latvia is rich in clay resources; therefore, this study uses readily available local resources. The use of local raw materials in construction reduces the environmental impact and reduces the cost of the finished product substantially [15].

Geopolymers are low-temperature ceramics with a high temperature resistance, relatively high mechanical strength, low elasticity module and shrinkage. However, these polymers are relatively fragile, like most ceramics.

[16]. Inorganic materials and wood are different materials. They both have the ability to absorb water easily but their different changes in form makes gluing difficult at variable humidity and temperature. People see them as cracks formed between the timber and the mineral material [17].

Although considerable researches on material binding mechanisms have been conducted, there is no single theory that can explain all the phenomenal binding mechanisms comprehensively. Adherence is a multidisciplinary science that includes surface chemistry, physics, rheology, polymer chemistry, mechanics of materials (including stress analysis), polymer physics, fracture analysis, etc. Due to the complexity and the changing understanding of this subject, it is difficult to describe the binding mechanism in simple terms [18]. Currently the main binding theories, which characterize the wood, are as follow: (a) mechanical penetration theory, (b) chemical-link theory, (c) weak boundary layer theory, and (d) theory of absorption. Although these theories are incomplete, they can help us to understand the phenomena that occur between two surfaces better. It allows us to choose the most appropriate method for optimal gluing of respective materials [18, 19].

Interaction between the adhesive and the wood is very important. When building a wood composite material with an inorganic binder, it is essential to have the wood particles fully covered with the binder [1]. One of the possibilities to improve the adhesion between the wood and the geopolymer is increasing of the glue spot stiffness. However, a firm, rigid joint becomes brittle and loses the flexibility that may be become a deficiency of the joint during operation [17].

Extensive researches have been conducted on binding of the wood with organic glues; however, there are only few studies on the wood and inorganic binders. There is even less information about binding of the wood with geopolymers.

It is necessary to find out the main affecting chemistry of inter layer between wood and geopolymer. The aim of the study is to specify the interaction between wood and inorganic matrix (geopolymer). The objectives are: 1) to estimate elementary processes at the interface that sticks two solid materials together: physical intermolecular interactions (Van der Waals forces, hydrogen bounds, ionic bonds) as different kind chemical bonds or as mechanical interlocking; 2) estimation of defects caused by physical and chemical stresses at the interface due to wood swelling and shrinking with changing humidity and 3) estimation of alumina / silica ratio of geopolymer on the penetration in wood structure.

Experimental

Materials

Softwood – spruce (Picea abies L.), pine (Pinus sylvestris L.), and hardwood – birch (Betula pendula) with no wood faults, size 200 × 80 × 20 mm. Samples are made with the glued surface in tangential/radial plane and cross-sectional plane. Before gluing, samples were conditioned in 50 % air relative humidity and 23 ^{o}C temperature, wood humidity 12 ± 0.5 %. Clay powder SIA „Ceplis" (Latvia, Auce municipality); particle size fraction ≤ 0.5 mm and ≤ 0.1 mm; heated in 700 ^{o}C temperature by obtaining a calcined clay. Sodium silicate solution (liquid glass), produced by SIA "LEANA". The specified mass of sodium oxide in sodium silicate solution is 0.00135 mol·g^{-1} and the determined content of the dry matter is 47 %.

Equipment

Zwic mechanical strength test equipment; x-ray CT scanner (manufactured at the Centre for X-ray Tomography of the University of Ghent); Memmer GmbH thermostat with mechanical ventilation; Nabertherm GmbH muffle furnace; Joo-Labor-Press LAP60 press; scales.

For establishing of the linkage between the wood and the geopolymer, a geopolymer bind from clay, sodium silicate and water with the following proportion shall be prepared, please, see **Table 1**. Mix the liquid glass with calcined clay and, if necessary, some water. Mix the mass for 15 minutes approximately, then spread the glue over both surfaces of the wood with a brush and connect the surfaces with a little push.

Keep samples in a press under pressure below 1 MPa. After removal of samples from the press, wrap the glued samples in cling film in order to restrict the wood drying and maintain a damp environment for the polymerization reaction, and store at 75^{o}C temperature for 24 h. For hardening of the binder, samples are stored at 20 ± 5 ^{o}C temperature for 28 days.

Compression-shear mechanical strength

Make three replicate samples (blanks) with each type of binder, from which 10 samples (n = 10) are later sawed and tested in the compression-shear test.

The mechanical strength between the wood and the geopolymer binder is determined by standard CEN/TS 13354:2003 for testing of the bonding quality of solid wood panels. Since this standard is rather intended for wooden shields or panels glued in several layers, some adjustments have to be made for studying of the binder. To obtain a seam of smooth thickness, glue two wood boards, size 200x80x20 mm, and, after hardening of the seam (28 days), saw the samples, size 50x40x20 mm according to the standard, required for the compression-shear test.

Make sure that the samples are not sawed from the blank edges; and the compression-shear samples shall not contain any wood faults. Before the compression-shear test, condition the samples in 60 % air relative humidity and 23^{o}C temperature. Determine the compression-shear mechanical strength of the samples according to standard CEN/TS 13354:2003.

Table 1. Composition of the prepared geopolymers.

Series	Ratios			Fraction size of clay, mm	Pressing time, h	Wood	Grain
	Sodium silicate solution	Calcined clay	H_2O				
S1-1.4	1.4	1	-	≤ 0.5	1	Spruce	parallel
S1-1.4U	1.4		0,2				
S1-1.75U	1.75		0,5				
S1-1.8	1.8		-				
S1-2.4	2.4		-				
S1-3.0	3		-				
S1-2EG	2	1	-	≤ 0.5	4	(Spruce, pine and birch)	end grain
S1-2.35EG	2.35						
S1-2.7EG	2.7						
S2-1.7	1.7	1	-	≤ 0.1	4	(Spruce, pine and birch)	parallel
S2-2	2						
S2-2.175	2.175						
S2-2.35	2.35						
S2-2.525	2.525						
S2-2.7	2.7						
S2-2EG	2	1	-	≤ 0.1	4	(Spruce, pine and birch)	end grain
S2-2.35EG	2.35						
S2-2.7EG	2.7						

Scanning with x-ray computed tomography

Make the samples required for scanning analogically to the samples for the compression-shear test. Make the samples using the universal circular saw machine and cut the small samples (0.8 and 2 for micron scanning) with a scalpel. For 15-micron scanning, prepare samples in size of 20×40×50 mm, 5-micron – 5×5×20 mm, 2-micron – 1×1×2 mm, and 0.8 micron sample – only about 0.01 mm^3. Scan with the x-ray computer tomography equipment. Adjust the equipment upon necessity for obtaining of a clear picture at the required zoom. A darker colour represents less dense areas, such as the cell cavities, and a lighter (white) colour shows more dense areas, such as the cell walls.

During assessment of the humidity impact on the binder, test four samples of the wood. First, scan the samples, then immerse them into water for 1 minute and scan again when dried.

Use descriptive statistics in the study to assess the validity of the results. Use the MS Excel program for calculations – determine the average arithmetic value (=AVERAGE) and the standard deviation (=STDEV).

Results and discussion

Mechanical strength in compression-shear test

In shear test, the S1 type binder shows a wide range of mechanical strength, for example, the mechanical strength of samples S1-1.8 is ranged from 0.44 to 3.53 MPa. The same applies to other binders, respectively: the strength of S1-1.4 ranges from 0.25 to 3.46 MPa, S1-2.4 – from 0.58 to 3.24 MPA, and S1-3.0 - from 0 to 1.47 MPa (one sample blank broke while sawing).

When comparing of the obtained strength with the strength determined in F. Gouny's study (determining of shear strength between the wood and the geopolymer brick), where the strength of 2 MPa at one composition of geopolymer and 1 MPa at the other composition of geopolymer was determined [20], it can be concluded that the obtained strength is near to it because the average strength of S1-2.4 is 1.89 MPa.

In the gluing process, it can be observed that the wood absorbs the moisture from the binders rapidly. Affected by the moisture, the wood moisture swells and curves thus destroying the seam that has not hardened yet mechanically. Pressing time 1 h is too short for evening of the humidity in the wood. The coarse particles (0.5 mm) of the applied clay penetrate in the wood during pressing, but after removing of the force the wood returns to the previous form and presses out the individual grains of clay. It destroys the seam that has not hardened yet mechanically.

The shear mechanical strength parameters of the improved binder S2 reach a better strength, however, the range of the data still remains wide. The maximum strength of S2 type binder can reach even 7.21 MPa (S2-2.35); at the same time, the same binder demonstrates a very low strength as well – only 0.29 MPA. It means that there is some binding ability between the wood and the geopolymer but there are some unknown factors that

Table 2. Mechanical strength of compression-shear test, MPa.

Series	Spruce			Pine			Birch		
	Average	STDVA	Max	Average	STDVA	Max	Average	STDVA	Max
S1-1.4	1.33	1.05	3.46	-	-	-	-	-	-
S1-1.4U	x	x	x	-	-	-	-	-	-
S1-1.75U	x	x	x	-	-	-	-	-	-
S1-1.8	1.49	1.13	3.53	-	-	-	-	-	-
S1-2.4	1.89	1.06	3.24	-	-	-	-	-	-
S1-3.0	0.96	0.37	1.47	-	-	-	-	-	-
S2-1.7	1.19	0.81	1.98	0.60	0.68	1.89	-	-	-
S2-2	3.30	1.41	5.39	1.27	0.83	3.08	-	-	-
S2-2.175	4.37	1.48	5.41	3.19	1.82	6.08	-	-	-
S2-2.35	3.57	2.38	7.21	2.66	1.70	5.72	2.13	0.99	3.78
S2-2.525	4.80	2.08	7.08	2.01	1.30	4.47	1.34	1.33	4.11
S2-2.7	3.68	0.90	5.22	2.52	2.19	6.73	1.16	1.05	2.91
S2-2EG	1.48	0.36	1.91	1.32	0.47	2.02	-	-	-
S2-2.35EG	1.21	0.43	1.88	1.16	0.33	1.80	-	-	-
S2-2.7EG	1.94	0.68	2.75	1.12	0.64	2.39	-	-	-

affect it considerably. S2-2.525 presents the average strength of 4.8 MPa, which is more than two times better than the one of S1 composition.

To achieve maximum binder penetration depth, gluing of wood with cross-section planes is applied. Theoretically, as the penetration depth increases, the mechanical strength should increase as well [18].

Both S1 and S2 type binder for assessing of the cross-sectional plane gluing strength was used. The S1 type binder showed negative results in both tree species. In this case, the S2 type binder shows in the compression-shear test the strength similar with S1 binder in plane. At ratio 2.7:1, the average strength reaches 1.94 MPa and the maximum strength reaches 2.75 MPa. A narrower range of data was observed; and, it might be caused by the insufficient cohesion of the geopolymer.

Comparing the species effect, the spruce wood shows higher shear strength parameters than the pine, and both are higher than birch. Similar to the parallel grain also end grain binding strength of spruce is higher than pine. Although, the maximum values are relatively close to the spruce, and S2-2.7 strength is only for 0.36 MPa lower than the one of spruce.

The wide range of data indicates to a high error factor in the glue seam. Referring to the theory of the weak boundary layers [21], it can be concluded that preventing or minimizing the defects in the glue seam and/or increasing the mechanical strength of the geopolymer, it would be possible to improve also the average shear strength between the wood and the geopolymer.

Visual observations

The binding capacity of the wood and the geopolymer may be characterized with mechanical strength, but it results in destruction of the glued wood sample. After the test, a destroyed sample of the wood and the force at which the sample was broken down remains. Examination of the destruction spot after the compression-shear test leads to conclusion that in general, the samples tear down through the binder **(see Figure 1 (A, B),** which might indicate to a weak cohesion of the binder.

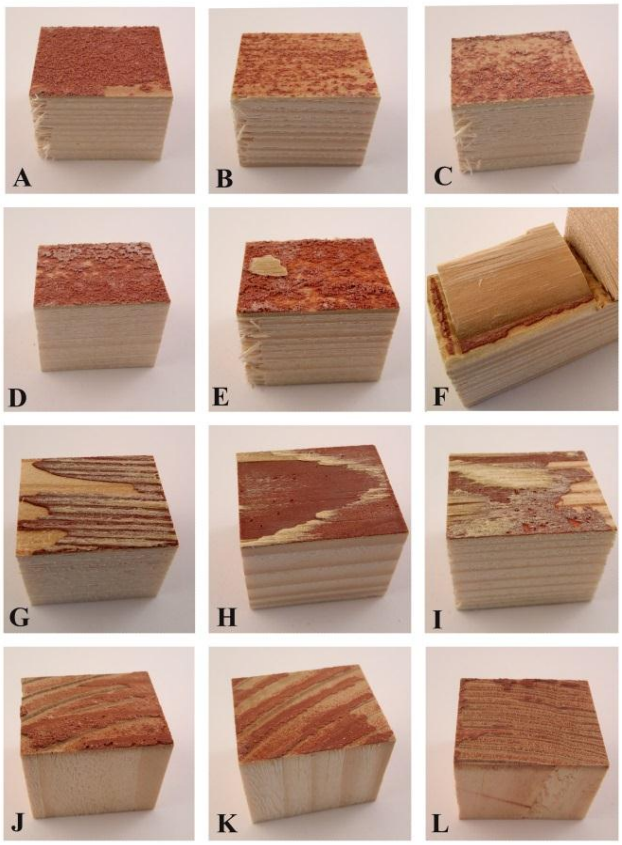

Fig. 1. Examination of the destruction type after the compression-shear test.

Some samples show also destruction through the border surface **(Fig. 1 (C, D),** which indicates to a low adhesion between the wood and the geopolymer. Although a complete destruction through the wood was not stated, in **Fig. 1 (E),** a partial destruction through the wood can be observed as well. This type of destruction is observed less frequently, however, it indicates to the possibility of sufficient adhesion.

The results of the compression-shear test and the observations of the joint destruction characteristics show that the possible causes are the too big clay particles, which do not allow application of a smooth glue layer; in addition, large clay grains may form micro-fractures in the binder after removal of the pressing load. Another possible cause is the impact of water on forming of geopolymerization links. Since the wood has good water absorption properties, it is possible that the water from the middle layer of the binder is drained first to the wood; therefore, the conditions required for polymerization reaction are not provided. The water creates environment for dissolving of alumosilicates and provides ion movement [10]. Whereas, the conditions required for polymerization reaction might be provided in area closer to the wood where the humidity level is higher. This would explain why the fracture goes mainly through the binder.

A similar analysis of the nature of the destroyed samples of S2 binders shows that the destruction through the wood **(Fig. 1 (F-I)),** or partial destruction through the wood is much more common, which could explain the improvement of strength indicators. S2 samples show glued seam defects before breaking as well; however, they are less common. Destruction through the binder was observed less frequently; however, it was typical to nearly all samples where the cross-sectional plane was glued. **Fig. 1 (J-L)** clearly shows that the geopolymer penetrates in the early wood very well but almost never in the late wood. X-ray computer tomography scanning of samples allows more extensive explanations of these observations.

X-ray computer tomography analysis

Results of the scanning did not show penetration of the geopolymer into cells. **Fig. 2** demonstrates the incomplete filling of open cells with the binder. Examination of the samples with various binders leads to conclusion that the calcinated clay particles contained by the geopolymer are too large to penetrate the cell wall. Despite sodium silicate cannot be shown separately in the scanning images, it can be assumed that it has a higher penetration ability, which is witnessed by the wood colour changes in the top layer (up to 1 mm). It cannot be detected, at which level geopolymer links between the penetrated liquid glass and the non-penetrated clay are establishing. **Fig. 2** also shows that the binder has closely backed to the wood which means that an unimpaired border surface can provide sufficient adhesion [21].

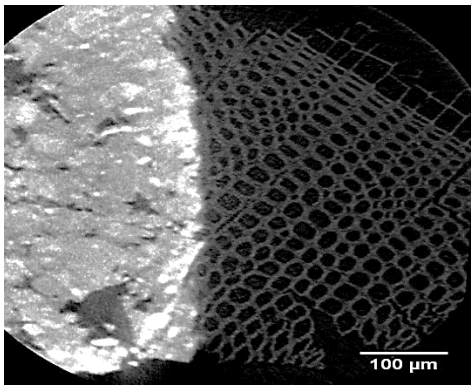

Fig. 2. Wood and geopolymer seam.

During examination of penetration/non-penetration of the binder into the wood, no significant differences in tree species were observed. Whereas penetration occurs in cut pores or uneven surface only, the surface roughness partially affected by the tree species is relevant. Comparing of early and late wood shows that in respect of pine and spruce, the penetration of the binder is better in early wood than in late wood, which can be explained by the difference in pore size in early and late wood and by increased content of lignin.

The resulting images explain the wide data range in the compression-shear test. **Fig. 3 (A)** shows the uneven structure of S1 binder layer very clearly. Although the average binder layer thickness is uniform, one can see the larger clay grains of the coarse fraction in sizes that reach the thickness of the layer. At the same time, there are large voids without the binder; and, it justifies the weak cohesion of the binder. One can see a thin, almost continuous binder layer along both sides of the wood border surfaces which suggests of sufficient adhesion. A better situation is observed in samples with S2 type binder. **Fig. 3 (B)** shows pine sample S2-2.175, where the binder forms a smooth seam and penetration of the binder in the cut trachea is visible.

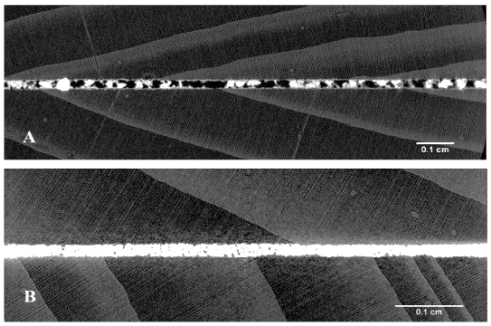

Fig. 3. Geopolymer binder seam – (A) S1-2.4 inhomogeneous binder seam; (B) S2-2.35 homogeneous binder seam.

Examining of the seam structure of different composition leads to conclusion that many small cracks are forming after hardening of the seam filled with binders with a smaller amount of sodium silicate, such as S2-1.7, and the binder looks like a dried lake bed.

Samples with a bigger amount of sodium silicate solution have large pores and long cracks. Whereas, the binders with the best strength have small dispersed small pores and small cracks. Formation of cracks and voids is caused by the geopolymer shrinking during drying. It can be concluded that the uneven layer structure after drying is one of the key factors affecting the mechanical strength of the samples. A uniform binder layer that is facilitated by an appropriate ratio between the sodium silicate solution and the calcined clay could provide a higher and more predictable strength of the seam.

Assessment of the moisture impact on a hardened seam shows that only one minute in the water causes additional cracks, which means reduction of the mechanical strength. Although the geopolymer is not soluble in water, it forms a hard and brittle glue seam that eliminates the mechanical strength during operation.

Conclusion

The binding ability between the wood and the geopolymer is sufficient; it can reach the compression-shear strength that exceeds 7 MPa. Spruce has the best strength - it reaches the compression-shear strength of 7.21 MPa. Birch has the weakest strength as it shows only 4.11 MPa of the compression-shear strength.

Sodium silicate solution and calcined clay mixtures in proportion ranged from 2.1:1 to 2.6:1 show the best binding properties.

Swelling and shrinking of the wood causes formation of cracks in the binder layer, which reduces the seam strength.

Aluminium/silicon proportion does not affect penetration of the binder in wood; instead, the seam strength is primarily affected by the binder seam homogeneity. Binding between the wood and the geopolymer is provided by the physical interaction between the molecules or some chemical links.

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