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Flame retardancy of Scots pine (Pinus sylvestris) by using polyethylene glycol 400 and phosphoric acid

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ABSTRACT—*Wood is natural material that can be used in many fields because it has good mechanical properties and easy to work with but its resistance to fire considered weak. Fire resistance is an important material characteristic in many applications. For this reason, a lot of research made on this topic for finding solutions in both physical and chemical ways, to increase fire resistance.This study presents experiments for improving the fire resistance of scots pine (Pinus sylvestris) by using Polyethylene glycol 400 and phosphoric acid as fire retardants. The investigation was made by using the single flame source test as fire test according to the standard (EN ISO 11925-2:2011). In addition, contact angle was measured to calculate the surface energy, surface polarity and wettability in order to give a better understanding to the used fire retardants and there effect on the surface of Scots pine. Results showed that Polyethylene glycol 400 and phosphoric acid are effective as fire retardants in case of a high dose of phosphoric acid and minor time in the microwave.*

Index Terms— *Fire resistance, Surface energy, Surface polarity, Wettability.*

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I. INTRODUCTION

Wood is considered the best material in building because it has better compatibility with different materials then the other construction materials such as concrete and steel. On the other hand, its mechanical properties are weaker. For compression strength, wood is weaker then concrete and for tensile strength, it is weaker then steel. In addition, its resistance to fire is considered weak [2]. The flames spread faster in wood compared to other conventional building materials but its strength is dropping slower compared to eg.Steel. Fire resistance is an important material characteristic in many applications. For this reason, a lot of research were made on this topic for finding solutions in both physical and chemical ways, to increase fire resistance.Fire retardants have different effects on different materials because each material has a certain response to fire and that goes back to many factors, which can be concluded in these questions: how easy is it to ignite the material, and how fast will it burn? Does the flame spread all over the surface? How rapidly do the flames infiltrate into a wall or barrier, how much and at what speed heat is released during the burning, how much smoke and toxic gas is generated, and finally, what is the speed of the chemical extinction of flames. However, first, we should understand the operation of fire retardant chemicals, the differences between fire retardants and how can choose which one is better to use depending onsituation [16]. The fire or flame-retardants are created to decrease the temperature of materials. When ignition occurs, the flame-retardants create thermal degradation, as they also raise the amount of char and reduce the flammability [3]. Fire retardants have two kinds of action: physical and chemical. For the physical action, there are many ways for delaying the ignition. Cooling is one of these features; there are some fire retardants, which can decrease the materials temperature. Coating is another way of delaying ignition where fire retardants can form a protective layer that prevents the underlying material from combusting. Dilution is the third way in which the retardants release water and carbon dioxide during burning. Each fire retardant has its better effect on a specific kind of material so the choice of fire retardant depends on the substrate and its unique set of characteristics.

Among others, phosphorus compounds are well known as fire retardant of wood [4-5-6]. The phosphorus compounds are efficient as fire retardants, because these reduces the thermal degradation of wood [6]. The phosphorus chemicals are forming acids which decrease the temperature of the wood [7] and as a result increase its dehydration and char formation [8]-[9]. Polyethylene is one of the fire retardants that showed up some years ago. Compared to other fire retardants Polyethylene is not well known as fire retardant. In 1995, Polyethylene was used in a research with phosphate as a fire retardant. The results were good but it showed up a problem when the temperature reached 80°C the compound that was used together in this research started to decompose and became less stable [26]. In 2016 a study was made about the influence of molecular weight of Polyethylene glycol (PEG) on thermal and fire protection of pentaerythritol phosphate (PEPA).Four types of PEG were used with different molecular weight PEG 150, PEG 200, PEG 400, PEG 600. The results of the fire protection of fire resistant coatings and the intumescence ratio test showed that PEG 600 has no efficiency on the fire resistance, but PEGs with low molecular weights are more efficient as fire retardants for the intumescent coating. Thermal degradation, results showed that char forming capability of intumescent coatings could be enhanced if PEG have low molecular weight [10]. In this study Polyethylene glycol with molecular weight 400 was used with phosphoric acid to enhance the fire resistance of Scots pine in order to introduce it later into the cement bonded particle board. Wood

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could have different features because of growth place, age and season of harvesting the wood. Because the sugar content and the extractives of wood are different forvarouis wood species to another [12]. It is important to choose the right wood species and the ratio of wood-cement and ratio of cement water because the amount of sugars and extractives has an inhibiting effect on the cement hydration process [11]. Scots pine is a popular species in the cement particleboard production because it contains few extractives. The aim of this paper is to find suitable fire retardant forScots pine in order to use it in the cement bonded particleboard production.

II. Materials and Methods

A conifer wood species was used, the Scots pine (*Pinus sylvestris*). 40 samples were used with dimension of (90x250x10). All samples were sanded with sand paper (grit size 120).As fire retardants Polyethylene glycol with molecular weight 400 (PEG400) and phosphoric acid were used with different ratios in order to help the phosphoric acid to react with the PEG 400.Wood samples were kept in a kitchen type micro wave with different time periods under temperature of 300°-350°C. Table 1 shows the different fire retardants dozes and time in the microwave.

Table 1: Time in microwave and amount of polyethylene glycol 400 and phosphoric acid for each sample.

	a	b	c	d
PEG (400) (g)	5	5	5	5
Phosphoric Acid (g)	1	1	2	2
time in microwave (s)	40	60	40	60

A. Surface modification

Wood samples were kept in room climate under temperature of 20°C and relative humidity of 60% for 24 hours until reaching moisture content equilibrium of 6%. After that surface of samples was treated with different amount of PEG400 and phosphoric acid (see Table 1) by painting the fire retardants with brush. After treating the surface, samples were put into microwave with different time in order to open the wood pores and help the fire retardants to react together. After that samples werekept in room climate at 20°C temperature and 60% humidity to dry for 2 weeks till reaching 12% MC.In most of the relevant publications the free surface energy was measured at 12%MC so the results of this work are comparable to the results of other publications [20]-[23]. Finally, fire test was made.

B. The single flame source test

This test is according to the standard reaction to fire tests. Ignitability of products subjected to direct impingement of flame. Part 2: Single-flame source test (ISO 11925-2:2011)with Taurus Instrument (Fig1). The aim of this test is to define ignitability of a vertically oriented test sample exposed to a small flame. The samples can be exposed on three different spots, either on surface, on edge, or on side. In our test, the surface was used. The samples dimension should be 250mm x 90mm, and for thickness, the maximum is 40 mm. For preparing the samples to this test, it should be marked on surface by two lines. The first line is 40 mm above the bottom of the

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sample and the second line is 150 mm higher than the first one. This marks off the flame area according to the standard. If the flame exceeds the second line, it is out of standard. The first line is where the flame starts. Time for the test is 30s. The first 15s is for burning and the second 15s is for watching if the samples will start to ignite by themselves or stop burning.

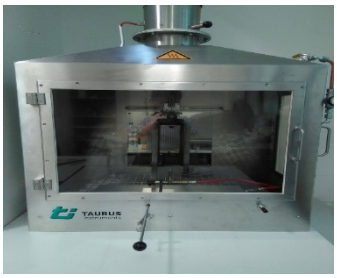

Figure 1: Taurus instrument

C. Contact angle measurement

Measuring contact angle of wood is the way to calculate its surface tension based on the Young-Dupré equation. Young (1805) declared that for a homogenous and ideally smooth surface the contact angle for a wetting droplet is written by the following equation:

$$cos\theta = \frac{\gamma sv - \gamma sl}{\gamma lv} \qquad (1)$$

Where γ_{sv} is the surface tension at the solid-vapour interface, γ_{sl} is the surface tension at solid- liquid interface and γ_{lv} is the surface tension at liquid-vapour interface.

The contact angle θ is formed between a drop of liquid (a demi sphere) relaxed on an ideally smooth solid surface and a tangential line is drawn to the drop in the point of intersection. (Fig.1). This is the reason for choosing a sanded surface as for this is the smoothest wood surface [24]-[25].

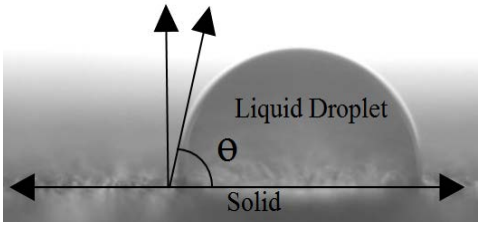

Figure 2: contact angle.

Wu [1] proposed that the summation of a dispersive component and poplar component results intermolecular energy between two materials. Generally, the surface free energy is proportional to the intermolecular energy [17].

There are two methodesto calculate the surface with polar and dispersive componenet the geometric mean equation and the harmonic mean equation. Fowkes equation is for two liquids method where one polar liquid and one dispersive liquidis used [22]. Distilled water is one of the most appropriate liquids to be used as polar component, and Diiodomethane as dispersive component [18]-[19]-[20]-[21].This is the reason of using distilled water and diiodomethane as test liquid for measuring the contact angle in this test. Contact angle Θ was measured with both distilled water and diiodomethane by PGX goniometer.Both water and diiodomethane used as droplet of 5 μL.The PGX goniometer has a digital camera connected to a software in computer taking the images every 0,1 second for 5 seconds.Themeasurement have been performed in dynamic mode, and the apparent contact angle was

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taken in the first second. The water surface tension was 72,8 mN/m. Water, polar component surface tension 46,4 mN/m and water, dispersive component surface tension 26,4 mN/m. 10 Measurement of contact angle per each sample was made on both normal and burning surface of the samples. Surface free energy of wood and also quoted surface free energy has been calculated upon Fowkes model [22] equation 2 and 3:

$$\gamma_S^D = \gamma_1/4)(COS\theta + 1)^2 \quad (2)$$

$$(\gamma_S^D \gamma_1^D)^{1/2} + (\gamma_S^P \gamma_1^P)^{1/2} = \gamma_1 (COS\theta + 1)/2 \quad (3)$$

With:

γ_S^D: Dispersion interaction.

γ_S^P: Polar interaction.

γ_1: Surface tension.

γ_1^D: Dispersive component surface tension.

γ_1^P: Polar component surface tension.

θ: Contact angle.

The increase in contact angle indicates the increase in hydrophobicity when the contact angle is measured using very hydrophilic liquid (i.e. water), since larger contact angle means less wetting of the surface by water [27]–[28].Low contact angle means good wettability, especially if the contact angle is lower or equal to 30° [29].

III. RESULTS AND DISCUSSION

A. The single flame source

Reaction to fire was tested according to standard. (*EN ISO 11925-2:2011*).All treated samples full-filled the standard requirement becausenone of the burning lengths did exceed 150mm (see Fig.3).

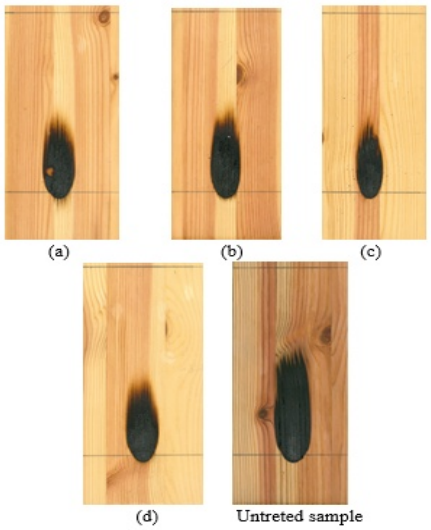

Figure 3: Results of single flame source test (a) is treatment a, (b) is treatment b, (c) is treatment c and (d) is treatment d.samples

Remarque: Samples in Fig.3 are picked randomlly among the replacation.

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As Fig.4 shown fire retardants were successful on improving the fire resistance of Scots pine compared to the untreated wood samples. In previous study it was found, that PEG with lower molecular weight than 600 is suitable as fire retardant [10]. In this study, PEG with molecular weight 400 was used and results proved that it is suitable as fire retardant because fire resistance of samples treated with treatment (a) increased by 6.25% while samples treated with treatment (b) increased by 12.5%. Samples treated with treatment (c) its fire resistance increased by 25% and samples treated with treatment (d) increased by 18.75%. For the results it appear that time in the microwave and the amount of the used phosphoric acid has significant effect on the performance of the fire retardancy of PEG 400 on scots pine.With the increasing amount ofthephosphoric acid,fire resistance increased becausephosphoric acid decreases temperature of wood [7] and increase dehydration and char formation [8]-[9].In other hand, the effect of time in micro wave is deferent because when 1g of phosphorus acid were used with PEG 400 the largest time in microwave gave the best fire retardancy while minor time in the microwave gave the best fire retardancy in case of larger amount of phosphorus acid which was 2g.In previous research phosphate was used with PEG it was found that when temperature exceeded 80°C chemicals decompose and became less stable [26]. This is the reason that fire retardants has low effect on the fire retardancy of wood in case of big amountof phosphoric acid because large time in microwaves results higher temperature which leads to decomposition between PEG 400 and phosphoric acid.Thatdidn’t happen when the amount of phosphoric acid became small.

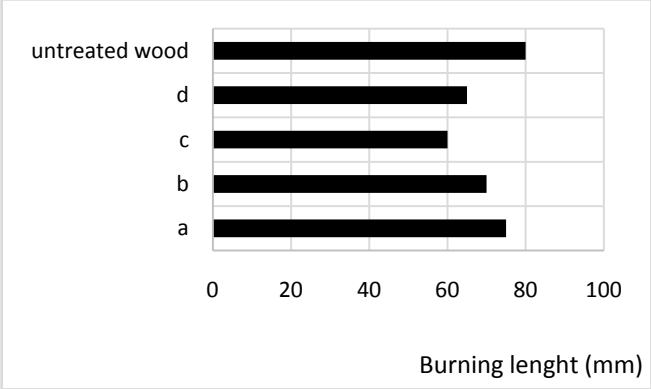

Figure 4: Burning lenght (mm)

B. Contact anglemeasurement

After finding the contact angle Ɵ, calculation was made to determine the surface energy [13], surface polarity [14-15] and surface wettability [16] as represented in Fig.4.Contact angle wasmeasured on both normal surface and burned surface (see Fig.5),inorder to understand the surface of the scots pine and the interaction between the wood surface and the applied fire retardants.

Figure 5: Normal and Burned surface surface of wood.

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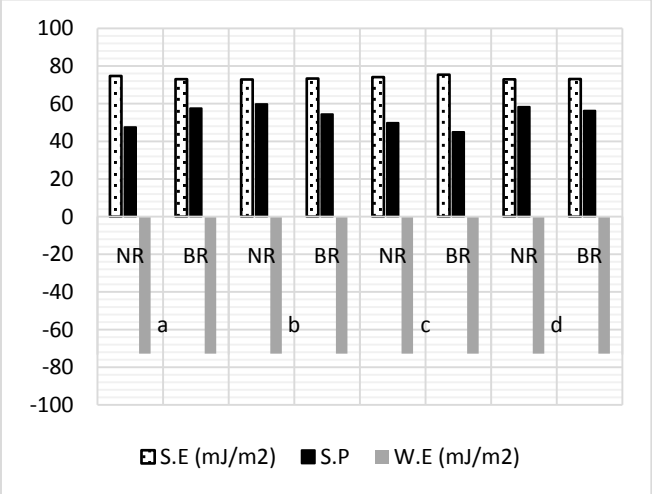

Fig.6total surface energy, surface polarity and wetting energy for normal surface (NR) and burned surface (BR) for all treatments (a, b, c, d).

As Fig.6 shows, all treated samples had the same wetting energy and all treated samples had negative wetting energy, which means chemicals could penetrate into the samples and gave good coating for the wood surface. The surface energy had a high result in all treatedsamples. There was no big difference in the surface energy of all treated surfaces. In other hand surface polarity had a lower results comparing to surface energy,which meansless char formation. All surfaces are polar or non-polar this is why the surface energy of materials should be calculated in most of studies with one polar and one non-polar liquid. Surface with polar molecules are usually easily hydrated by water..Sample treated with treatment (c)had the lowest surface polarity among all treated samples .Surface polarity of the burned surface is lower than the surface polarity of the normal surface except for samples treated with treatment (a).

IV. CONCLUSION

PEG 400 and phosphoric acid had influence on the surface polarity of the wood with large amout of phosphoric acid mixed with PEG 400. Shorttime in microwave can decrease the surface polarity of the wood. PEG 400 and phosphoric acid are suitable fire retardant for scots pine and can be introduce it into the cement bonded particleboard production but only after investigating the fire retardants influence on the cement hydration.

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