Article

Influence of Copper and Biopolymer/Saqez Resin on the Properties of Poplar Wood

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Abstract: There is an increasing interest in applying environmentally-friendly materials in wood protection technology. This include the use of less toxic active ingredients, as well as better fixation. This study investigates the formulation based on the combination of copper and Saqez resin on the physical and biological resistance properties of poplar wood. Samples were treated by either copper-ethanolamine (Cu/MEA) and/or Saqez resin at various treatment levels. A vacuum pressure procedure was applied. The retention, weight percent gain, water absorption, volumetric swelling, and decay resistance of the samples were then determined. The highest retention and weight percent gain were obtained at the samples treated with the combination of copper-based system and Saqez resin. Additionally, the combination of the copper and Saqez improved the physical properties and decay-resistance against white-rot fungus *Trametes versicolor*.

Keywords: Saqez; Copper; Physical properties; Biological resistance; Performance

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1. Introduction

Wood, as a versatile biocomposite material has a wide variety of applications such as furniture, construct structures, artifacts, tool making for several centuries [1]. However, the application of less durable wood species in outdoor applications is frequently limited [2]. Namely, wood is susceptible to decay by wood-destroying organisms such as fungi and insects. Predominant hygroscopicity, resulting from the presence of the abundant hydroxyl groups in wood renders it susceptible to fungal infestation. Wood and wood-based products often need treatments with preservatives in order to increase durability and achieve desired service life [3-5]. One effective method to improve the bio-resistance features of wood and wood-based products is the utilization of natural substances such as plant essential oils and resins [6, 7]. There is an ever-increasing interest in the application of essential oils due to their natural safety and environmentally friendly aspects. They are effective against bacteria [8, 9], fungi and moulds [10-14] and insects [15, 16]. Su et al. [17] found that the essential oils of *Eucalyptus citriodora* can prevent the growth of fungi and moulds. Yang and Clausen [18] obtained mould inhibition on southern yellow pine by geranium and thyme oil. Mohareb et al. [19] monitored the antifungal activity of 18 essential oils from Egyptian plants against wood-decay fungi and concluded that the essential oils of *Cupressus sempervirens*, *Citrus limon*, *Thuja occidentalis*, *Schinus molle*, *A. monosperma* and *Pelargonium graveolens* were the most potent inhibitors against fungi. Bahmani and Schmidt [20] studied 16 essential oils against wood decay fungi and moulds and found the lavender oil, lemongrass oil, and thyme oil to be the most effective against mould and wood decay fungi. However, although these oils improves the overall performance of wood, they are hardly classified as wood preservatives, as biocides have to pass strict efficacy and eco-toxicity assessment, prescribed by authorization agencies such as ECHA in EU.

Nevertheless, the use of plant essential oils in wood protection have some limitations. Implications of the natural oils is restricted to indoor and outdoor applications due to their high volatility, insufficient fixation, and leaching from treated wood if the oils are not combined with fixatives. Conjunction of natural oils with other biocides such as copper compounds are proposed for overcoming deficiencies of environmentally friendly preservatives such as natural oils and may be

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able to improve its efficacy against fungi and insects. Alfredsen and Flaete [21] obtained tall oil combined with copper can provide some protection for treated wood. Can and Sivrikaya [22] investigated the combination treatment of tall oil and linseed and copper on some properties of pine wood. They concluded that copper and oil combination could improve the physical and biological properties of wood as well as decrease copper leaching.

Copper (Cu) is one of the most effective and extensively used wood biocide. It can be effective against wood-decaying fungi and some termites if applied in sufficient quantities [23]. Copper-based preservatives are traditionally applied for the protection of posts, bridges, vineyard poles, noise barriers, fences, etc. In the past, copper was mixed with chromium compounds in chromated copper arsenate compounds (CCA). Nowadays, the CCA treatment is forbidden in Europe and the US for many applications mainly due to the end of life issues. The alternatives to these biocides are various organic-based substances such as ammoniacal copper quaternary (ACQ) copper azole (CA), and bis-(N-cyclohexyldiazeniumdioxy)-copper (CuHDo) [24]. Copper-based preservatives are usually combined with ethanolamine to improve fixation and enable better penetration [25]. Copper Azole preservatives are one of the most important solutions for wood preservation [26]. It is commercially available for residential applications, free of arsenic and chromium and able to apply for above-ground, ground contact, and freshwater applications. However, high levels of copper and relatively high formulation cost [27] as well as copper release and disposal issues in the treated wood, are recognized to be the main limitation for future use [28]. Moreover, Copper azole belongs to water-borne preservatives, and treated wood suffers from poor dimensional stability [29]. To enhance the water repellent properties of copper Azole-treated wood adding hydrophobic materials such as rosin solutions, coating compounds etc. are needed.

One of the most economically important non-wood forest products of Zagros forests in the west of Iran is pistachio tree resin known as 'Saqez'. Saqez is used for a wide variety of industrial and traditional applications such as food and pharmaceutical formulation [30]. Saqez is a semi-dense, adhesive, liquid resin comprising antioxidants, has antimicrobial, antibacterial, and antifungal properties and is applied in the preparation of ointments for skin disorders [31, 32]. The increasing request in wood protection technology for safe, low cost, sustainable, environmental, natural, and

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biodegradable for preservatives has convinced scientists and technologists to examine for a new source of natural materials, as substitutes for chemical ones. The present study aimed to investigate the impact of pistachio tree resin (Saqez) alone or combined with copper-ethanolamine (Cu/MEA) and Saqez on the physical and biological properties of poplar treated wood.

2. Material and Methods

2.1. Material

The boards were cut from the trunks of three 23-year-old poplar trees (*Populus deltoids*) located in the Khirod area (Mazandaran province, Northern part of Iran) and air-dried. Wood samples were cut from the boards for the various tests. All samples were free of defects such as knots, resin pockets and moulds and fungal infections. The wood sample sizes for the physical tests were $20_l \times 20_r \times 20_t$ mm^3 according to ISO 13061:2016 [33]. Five replicates were cut for each test, along with five replicate control samples. For fungal durability studies, the sample size was $50_l \times 25_r \times 15_t$ mm^3 [34] with five replicates. Before treatment, samples were oven-dried (103 °C, 24 h), to obtain constant moisture content and to determine their initial masses. The density of the oven-dried specimens was in the range of (460±20) kg/m^3. To ensure maximum uptake of the treatment solutions, none of the surfaces of the wood samples was sealed. The copper-containing wood preservative used in this study was copper-ethanolamine (Cu/MEA) consisted of $CuCO_3$ (Merck) and ethanolamine (C_2H_7NO). The resin of wild pistachio (*Pistacia atlantica*) trees called 'Saqez' was obtained from the area is located in Charmahal and Backtiari province, Southwest of Iran.

2.2 Methods

Before the treatment process, samples were divided into three groups. The first group was treated with copper-ethanolamine (Cu/MEA). The copper concentration was 0.5 % in all tests. This concentration is usually sufficient for the protection of wood in-ground applications. The second group was treated with various concentrations of (5, 10 and 15%) pistachio tree resin. Ethanol with 96% purity was used in the preparation of the resin. The Third group initially impregnated with

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copper amine based wood preservative, afterwards treated with resin at the concentration of 15% (Table 1).

For treatments, a vacuum pressure impregnation were applied in a pilot plant, whereby the vacuum of 0.8 bar lasted for 30 min, followed by pressure at 4 bar for 120 min. After that, samples were stored for two weeks in the lab ambience for evaporation of the solvent. The retentions for each treatment were calculated according to the Equation (1):

$$R = \frac{G \times C}{V} \times 100 \text{ kg m}^{-3} \quad (1)$$

G is the amount of treating solution absorbed the samples (kg); *C* is the concentration of the solution (%), and *V* is the volume of the samples (m^3).

After treatment, the oven-dry (103°C for 24 h) weight of all samples was evaluated and used to calculate the weight percent gain (WPG) (Equation 2):

$$WPG(\%) = \frac{M_2 - M_1}{M_1} \times 100 \quad (2)$$

Where M_2 is the sample weight after treatment, and M_1 is the sample weight before treatment

Table 1. Experimental design of the study

Test groups		Preservative solution (%)	
		Cu (%)	Saqez Resin (%)
A	Control	0	0
B	Copper/MEA	0/5	0
		0	5
C	Saqez	0	10
		0	15
D	Copper/MEA+Saqez	0/5	15

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2.2.1 Water absorption and volumetric swelling measurements

Water **absorption and** volumetric swelling tests were carried out by soaking the treated and untreated samples for 2, 4, 6, 8, 24 h and repeated at 24 h intervals at room temperature following ISO 13061:2016 [33]. The water was replaced with fresh one. **Water absorption and** volumetric swelling were evaluated according to the Equation 3 and 4, respectively:

$$WA(\%) = \frac{(W_1 - W_0)}{W_0} \times 100 \quad (3)$$

Where, *WA* is water absorption (%), W_1 is the weight of samples after immersion; W_0 is the oven-dried weight before immersion.

$$VS(\%) = \frac{(V_1 - V_0)}{V_0} \times 100 \quad (4)$$

Where *VS* is volume swelling (%), V_1 is the volume of samples after immersion and V_0 is the volume of samples before immersion.

2.2.2 Biological durability

Biological durability was evaluated on both of leached and unleached samples in accordance with European method [34] to assess the resistance to white-rot fungus. The decay chambers were Petri dishes containing 4.8% malt extract agar (Merck, Darmstadt, Germany) that were inoculated with an agar plug cut from an actively growing culture of *Trametes versicolor* (L.: Fr.) Pilát , strain CTBA 863 from France, derived from the strain collection of the University of Hamburg. Wood samples were oven dried at 103±3 °C for 24 h and weighed before fungal exposure. The treated and untreated samples were subjected to the respective fungi by placing them on mycelia grown in the Petri dishes. The wood samples were incubated for 16 weeks at 23°C and 65 ± 5% relative humidity. At the end of exposure time, the mycelia coverings on the sample surfaces were removed and weighed. After exposure, surface mycelium was scraped off and wood samples were dried at 103 °C for 24 h and weighed again to calculate the mass loss according to the Equation 5:

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$$ML(\%) = \frac{M_0 - M_1}{M_0} \times 100 \quad (5)$$

Where, *ML* is the mass loss (%), M_0 is the oven dry weight of sample before fungi test (g) and M_1 is the oven dry weight after fungi test (g).

2.2.3. Statistical Analysis

The obtained data were subjected to the analysis of variance (ANOVA). Means were analyzed and grouped using Duncan's Multiple Range Test (DMRT).

3. Result and discussion

Populus deltoids was chosen for this study because it is prevalently used in a wide variety of applications in Asia and Europe. The retention of preservatives is the first substantial factor that shows the quality of the impregnation. Retention of the active ingredients increases with increasing resin concentration (Figure 1). Namely, wood treated with the lowest concentration of resin retains 18.4 kg/m^3 of the resin while the samples that were treated with the highest concentration of the resin system, uptakes 74.5 kg/m^3 of the resin. It seems like that ethanol solution has sufficient viscosity that enables good penetration into wood. ANOVA test results indicated that there is a significant difference between the retention of preservatives at the 99% confidence interval ($P<0.01$). Duncan's test classified the value of retentions in five separate groups.

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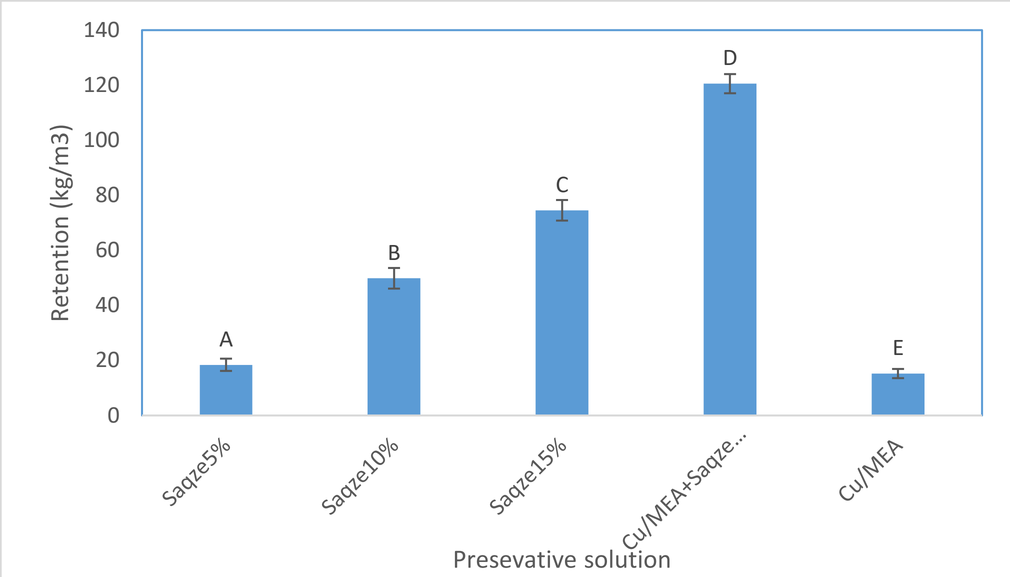

Figure 1. Preservative retention in the treated wood samples

The highest retentions were observed at samples that were first treated with copper-based system and in the later stage with resin-based system. These kinds of impregnations are called dual treatments and are reported in the literature already [35]. Retention of wax into copper-ethanolamine based system was higher than at non-copper treated wood. It can be presumed that one of the possible reason for this occurrence, could be assigned to wood swelling efficacy of ethanolamine [36].

Wood retention data are in line with WPG data. In general WPG increases with increasing resin concentration. Similarly, as reported for retention data, the highest WPG was determined at samples treated with copper-ethanolamine solution subsequently treated with resin (Figure 2). High WPG can be ascribed to the reaction of copper-ethanolamine complexes with wood and resin [37]. However, negative WPG was determined at samples treated with the lowest concentration of the resin. This can be ascribed to the fact that there might be some extractives leached from wood during impregnation with ethanol-based solution. Besides, part of the resin might evaporate from wood during oven drying. Predominately, as the melting point of the resin is rather low. Melting point is between 50°C and 60°C. ANOVA test results showed that there is a significant difference between the WPG of samples ($P<0.01$). Duncan's test classified the value of WPG in five groups.

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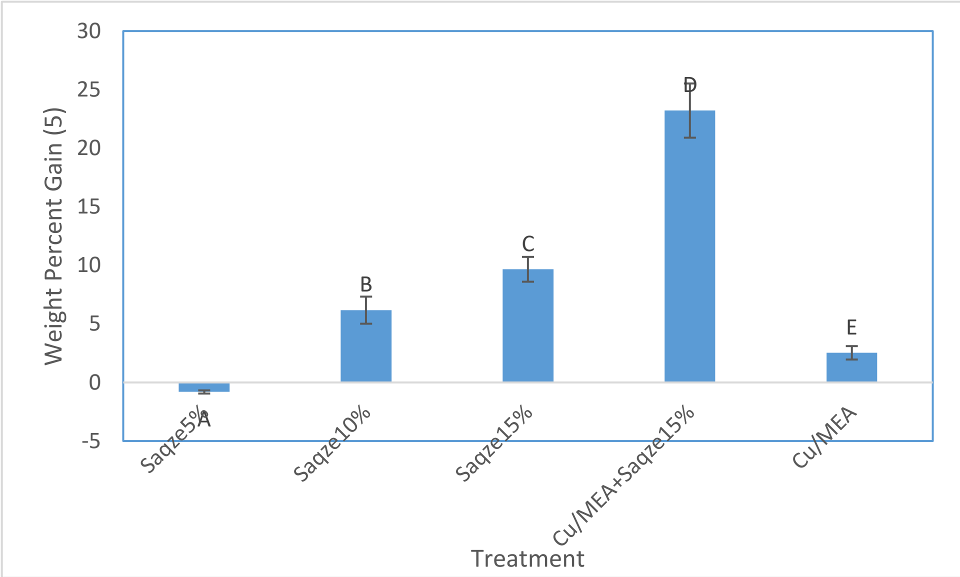

Figure 2. Weight percent gain values in the wood samples

Recent studies clearly indicate, that performance of wood in above-ground outdoor applications is a function of inherent durability (presence of biologically active extractives and/or fungicides) and water exclusion efficacy (the ability of wood to remain dry [38]. Therefore, studies of water ability are essential. Also, water repellents can slow down copper leaching [39]. As can be resolved from Figure 3, resin impregnation has a positive effect on water performance. Wood treated with resins uptakes less water than control wood. This can be ascribed to the lumen filling as well as to hydrophobic nature of the resins. A rather notable difference is evident from the beginning. Water repellency increases with increasing retention. Moisture content of wood treated with 5% concentration of resin was 25.2% while the MC of the samples treated with copper and 15% ethanol concentration of Saqez resulted in MC of 14.1%.

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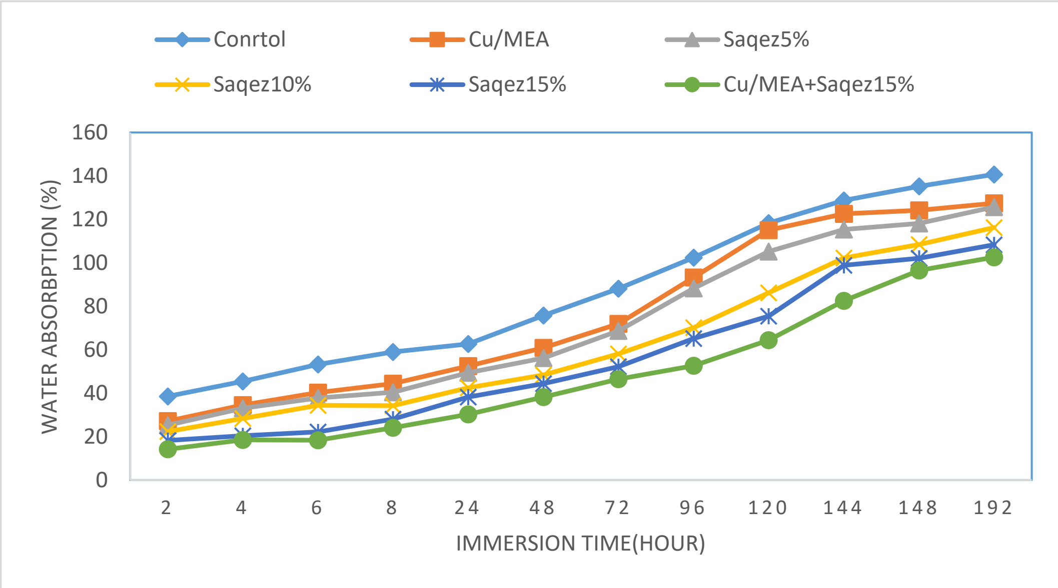

Figure 3. Water absorption in the wood samples

Figure 4 shows a good correlation between volumetric swelling and wood MC. What that exhibited good water exclusion efficacy exhibited good dimensional stability. Influence of the retention on wood swelling is even more prominent than on wood moisture content. In contrast to wood MC, the differences between treatments increases with immersion time. Thus, the highest difference was noted at the end of the 192 h lasting immersion. Namely, swelling at control samples was 26.7%, while swelling at samples treated with a combination of copper and Saqez resin resulted in swelling of 13.3%.

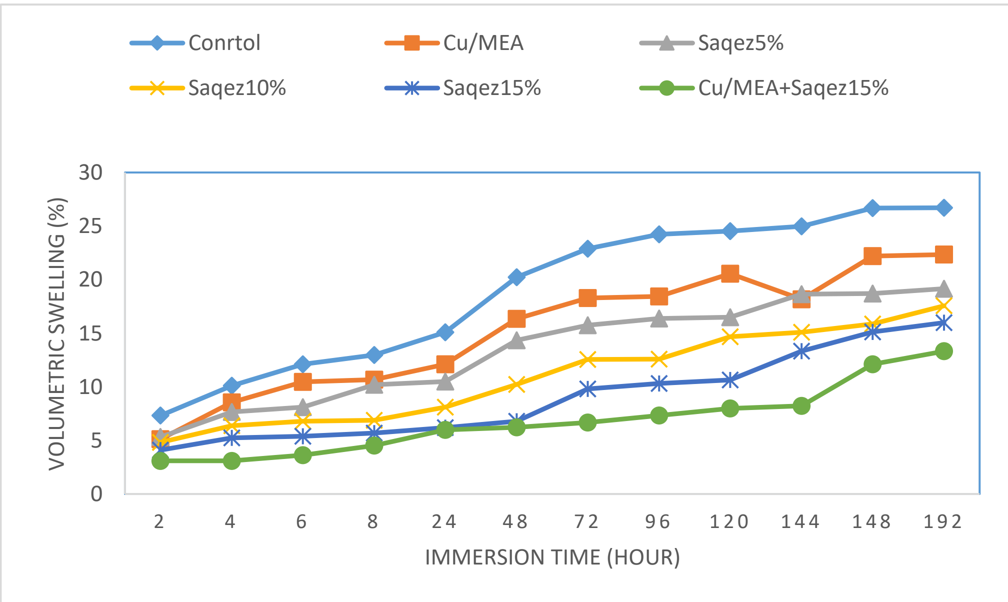

Figure 4. Volumetric swelling in the wood samples

One of the prime objectives of this study was to determine the performance of the respective treatment against wood decay fungi. Samples were exposed to white-rot fungi. White rot fungi are the most important organisms degrading the hardwoods [40]. Mass loss of the leached and unleached samples was rather high, ranging between 39.3% at non-leached and 44.8% at leached specimens (Figure 5). These data are in line with low durability classification of poplar wood [41]. Resins have limited on the fungi. Mass loss of the Saqez treated samples was a bit lower than the mass loss of the controls. The efficacy of the resin increases with increasing concentration of resin in the treatment solution. For example, mass loss of the wood treated with 5% ethanol solution of Saqez was 30.1%, whereas the mass loss of the wood treated with a similar solution with three times higher concentration was 20.4%. This mass loss is higher than the mass loss of the comparable oil or wax-based systems ([11, 42]). The major constituents in the Saqez are α-pinene (60.15%), β-pinene (8.68%), and α-terpinene (3.94%) [43-44]. The antifungal activity of Saqez resin could be related to the respective components. Antifungal activity of α-pinene, β-pinene and α-terpinene inicated in the previous study [45-47].

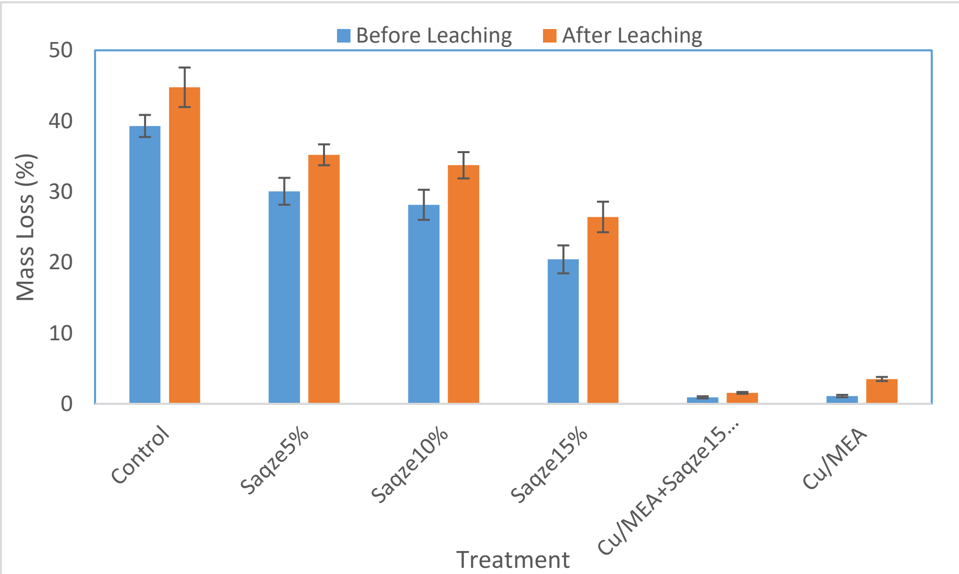

Figure 5. Mass Loss of wood samples subjected to *Trametes versicolor*

Presence of copper in wood slowed down wood decay considerably. Leaching reduces the influence of copper-based system, predominately as there were no secondary fungicides present [48]. However, as can be seen from the comparison of mass losses of leached and non-leached copper and copper-Saqez treated wood, a combination of copper and Saqez acted synergistically. At the very moment, we cannot conclude whether this is because of the limited effect of Saqez resin on the fungi, or due to the limited copper leaching.

4. Conclusion

Synergistic effect between copper and Saqez was investigated. Saqez has positive influence on water performance. Wood treated with Saqez exhibited better water performance, which reflects in better dimensional stability and likely limits copper leaching as well. Impregnation of wood with Saqez was not sufficient to limit fungal decay. However, the combination of copper-ethanolamine based wood preservatives with Saqez has a positive effect on copper efficacy against wood decay fungi.

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