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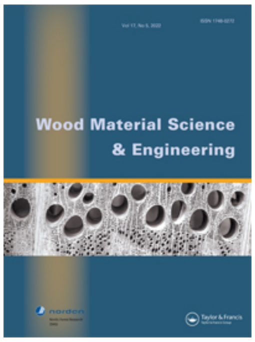

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Effect of nine-year soil contact on physical performance of crude tall oil impregnated, copper salt impregnated, and non-treated Scots pine posts

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ABSTRACT
Crude tall oil (CTO), a side product of sulphate pulping, has promising results in moisture and decay resistance. This article reports the Pilodyn indentation hardness, basic density, and mechanical performance of 600-mm long Scots pine posts after a nine-year soil contact test. The material consisted of round and square-shaped CTO-impregnated posts, supplemented by copper salt-impregnated and untreated reference posts. In total, 240 posts were installed in fertile soil in south-eastern Finland in spring 2010. Posts were harvested in May 2019. At the end of the test, 192 posts were in a condition that allowed the preparation of specimens for density and compression tests. Discs were sawn from the ground level, as well as above and below the ground level to measure the density and deformation under static 45,000 N parallel-to-the-grain compression load. Altogether 80% of the untreated control posts were rejected from further tests due to severe decay. Square-shaped control posts had a slightly higher survival rate than the round ones. After a nine-year soil contact, the performance of CTO impregnated. Scots pine posts were clearly better than that of untreated control posts and comparable to that of copper salt-impregnated posts. The study suggests that CTO has a high potential as a non-biocide wood preservative even in structures in soil contact.

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Crude tall oil; durability; impregnation; preservation; soil contact test

1. Introduction

Exposure of wood to moisture enables the growth of microorganisms, which erodes the surface and eventually deteriorates the wood properties by degradation of the cell walls. If not possible to use naturally durable wood species or assortments, such as heartwood, it is necessary to protect wood by physical protection (covering from liquid water exposure, surface treatment), impregnation, or modification techniques (e.g. Cogulet *et al*. 2018). Heartwood of many species, including Scots pine, is relatively decay resistant because of high concentration of stilbenes (pinosylvin, pinosylvin monomethyl ether, pinosylvin dimethyl ether) and terpenoids (e.g. Hart and Shrimpton 1979, Langenheim 1994, Harju *et al*. 2003, Venäläinen *et al*. 2003, 2004, Heijari *et al*. 2005).

Long-term outdoor exposure removes hydrophobic lignin from the fibre surface, exposing cellulose and hemicellulose containing hydroxyl (-OH) groups and weakening the hydrogen bonds (e.g. Laine *et al*. 1994). Hemicelluloses deteriorate more rapidly than cellulose (Green and Highley 1997). The moisture content of wood typically varies considerably over exposure time, causing dimensional changes and cracks that allow moisture and ultraviolet radiation to access deeper in wood. Depending on wood species, the minimum moisture content for microbial growth varies between 20% and 32% (e.g. Shmulsky and Jones 2011).

Most classic wood preservatives are based on the effects of toxic chemicals containing creosote, arsenic, chromium, zinc, or copper, which are biocides, i.e. harmful to living organisms (e.g. Zartarian *et al*. 2006, Wan 2013, Civardi *et al*. 2015) not only during their production and service life, but also after the primary life cycle. Due to environmental concerns and health risks, the use of biocides is getting more controlled, and their production is expected to gradually decrease worldwide.

Extractives are cell wall chemicals consisting of fats, fatty acids, phenols, terpenes, steroids, resin acids, waxes, etc. and they play an important role in the colour, smell, and durability of wood (e.g. Rowell 2005). Bio-oils are antifungal and increase the hydrophobicity of wood (e.g. Lourençon *et al*. 2016). Crude tall oil (CTO), the primary source of which is pine tree ("tall" is the Swedish word for "pine"), is a side product of sulphate pulp production. CTO is a viscous and sticky dark brown liquid chemically composed of 38–53 wt.% fatty acids, 38–53 wt.% rosin acids, and 6.5–20 wt.% unsaponified (neutral) compounds (Aro and Fatehi 2017). The oil composition varies between pine species, geographical locations, age of tree and wood material, and pulping conditions. The average yield of CTO is 30–50 kg per ton of pulp.

CTO deserves attention as a wood preservative because it is environmentally harmless, available in large quantities, and

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has a relatively low price in comparison to most other potential wood preservatives. Without further refining, CTO is non-biocidic, while its wood protection effect is based on forming a physical barrier against moisture. Several studies have been carried out on the application of pine tree extractives for wood protection (e.g. Passialis and Voulgaridis 1999, Hyvönen *et al*. 2006, Nemli *et al*. 2006, Koski and Ahonen 2008, Temiz *et al*. 2008, Möttönen *et al*. 2012, Heräjärvi *et al*. 2014). However, there are no public results on the effects of CTO impregnation on the performance of wood in a long-term soil contact. This article reports the durability performance of CTO-impregnated, copper salt-impregnated, and untreated control posts made of Scots pine (*Pinus sylvestris* L.) after a nine-year soil contact exposure. Round and square-shaped posts, both with two different diameters commonly used in fencing and similar applications, were analysed in terms of density, Pilodyn indentation hardness, and resistance against static parallel-to-the-grain compression.

2. Materials and methods

The materials originated from thinning stands of Scots pine in south-eastern Finland, harvested in winter, early 2009. The trees were relatively young (<25 years, on average), thus the trunks consisted almost completely of juvenile wood and sapwood. The round posts (two diameters: 80 and 120 mm) were rotary peeled and square posts (two dimensions: 50 × 50 mm and 100 × 100 mm) were sawn into their dimensions, and subsequently impregnated during the year 2009. Two ambient pressure hot oil bath CTO impregnation treatments, followed by a curing stage at a maximum temperature of 160 degrees Celsius, were used. The difference between the two treatments was the viscosity of the oil. Metal tags with identification codes were screwed on the top side of each post. The posts were treated as follows:

(1) CTOI: crude tall oil impregnation using the process of Ekopine Ltd. Average dry weight gain 215 kg/m^3 (dry matter based)
(2) CTOII: crude tall oil impregnation using the process of Ekopine Ltd. Average weight gain 270 kg/m^3 (dry matter based)
(3) Copper salt: commercial copper-chromium-based pressure impregnation to NTR AB class
(4) Untreated control

The numbers of copper salt, CTO I- and CTO II-impregnated and untreated control posts in the soil contact test were 60, 60, 60, and 10, respectively. In addition, 10–20 reference posts from each treatment were stored indoors in dry conditions; this reference group is called the "non-exposed posts". The exposed posts were implanted on a former agricultural field in Punkaharju, south-eastern Finland, in summer 2010. The 600 mm long posts were dug into the ground so that 200 mm was underground and 400 mm was above ground. Woody plants were cleared from the site regularly, but grasses and other annual plants grew on the site, partly affecting the above-ground conditions of the posts, mainly during the late summer season.

Posts were harvested on May 27, 2019. They were first cleaned gently to remove the soil particles, grass, mould, etc., using a wire brush and chisel. The discs for density and compression resistance tests were sawn from three different positions: ground level, above ground level, and below ground level. Vertical location of each disc was adjusted so that defect-free specimens (no knots or excessive decay) could be prepared. The top and bottom discs were, however, prepared from at least 150 mm distance from the post ends. The thicknesses of density and compression resistance specimens were 20 and 40 mm, respectively, while their crosscut area corresponded to the crosscut of the original post.

Pilodyn indentation hardness tests with Pilodyn 6J Forest (PROCEQ, Zurich, Switzerland) instrument and needle diameter of 1 mm were carried out after the exposure period. The test was performed by two needle hits close to the ground level position on two sides/faces of the post.

After the sawing and coding, the basic density specimens were soaked in tap water at room temperature. The soaking time was 7–8 days, after which the specimens were assumed to be fully swollen. Their green volume was measured by the water displacement method before drying at 105 degrees Celsius until a stable mass. The average oven drying time was 42 h. Basic density was calculated by dividing the dry mass by the green volume. In the case of CTO-treated specimens, oil leaching was evaluated visually both after the water soaking and after oven drying.

We were interested in the gross mechanical performance of a post with all defects. However, because there were considerable differences between the decay rates of above- and underground-level parts of the post, for example bending test would not have been particularly useful. Therefore, we decided to assess the longitudinal compression deformation of 40-mm thick discs from different vertical locations within the post. The discs represented the entire crosscut section of the post. After the sawing, cleaning and coding, the compression test discs were stored in a normal climate chamber (RH: 65 ± 3%, T: 20 ± 2°C) for approximately three months, during which time their mass was repeatedly measured. The equilibrium moisture content EMC was expected to be reached when the mass difference between two consecutive measurements was less than 0.1%. Before the parallel-to-the-grain compression test with Zwick/Roell Z050 material testing device, the dimensions of the specimens were measured with a calliper to calculate the surface area. In addition, the number and approximate area of checks was recorded. Discs were then compressed, and the resulting deformation was recorded. The load was applied with a constant speed of 1 mm/min up to 45,000 N and then released. The same procedure was applied for all specimens independently from their shape, size, or cracking.

SPSS Statistics software version 25 was used for statistical analyses. One-way ANOVA was used to compare the means (Pilodyn indentation depth, basic density, compression deformation) of more than two groups (specimen's vertical position, size, and treatment), followed by Tukey HSD Post Hoc test in case of equal variance assumed and Games-Howell test in case of equal variance not assumed. In case of normality assumption violation, Kruskal–Wallis test was conducted, followed by pairwise comparison. An independent sample *t*-test (parametric) and Mann–Whitney *U* test (non-parametric) was conducted to compare the differences between the two groups (in the case of crosscut shape and exposure group). Furthermore, Pearson correlations were calculated between Pilodyn indentation depth, basic density, and compression deformation.

3. Results and discussion

3.1. Survival rate

The most severely decayed posts or post parts were rejected from all further tests. As much as 80% of the untreated control posts and 6.7% of CTO I- and CTO II-impregnated posts were excluded, while all copper salt-impregnated posts were included. Of the untreated control posts, all 80 mm diameter round ones, 86.7% of the 120 mm diameter round ones, and 66.7% of the 50 × 50 mm and 100 × 100 mm ones were rejected from specimen preparation due to thorough decaying. Round control posts had, thus, higher rejection rate than the square-shaped ones. The cross-cut shape of the post, *per se*, is unlikely the actual reason behind the differences in durability. It is more probable that the quite robust peeled surface of the round posts was more susceptible to biodegradation than the sawn surface of square-shaped posts. In addition, the active surface areas as well as area-to-volume ratios differ between the post shapes.

In many cases, specimens were only prepared from the upper parts of the posts, while the lower parts were too badly decayed. The percentages of rejected density specimens at the underground, ground level, and top positions were 17, 8 and 2 for CTO I, 17, 3 and 2 for CTO II, and 92, 93 and 87 for control posts, respectively. The percentages of rejected compression test specimens were almost identical to the percentages of rejected density specimens.

The effectiveness order of different treatments against mass loss was: copper salt impregnated > crude tall oil > controls. The CTO-treated specimens absorb less moisture and dry faster than the copper salt-treated and untreated specimens (Hyvönen *et al.* 2006, Heräjärvi *et al.* 2014, see also Hosseinpourpia *et al.* (2020) for high-density fibreboard panels), therefore, preventing or slowing down the activity of decaying microorganisms. Because of the dry storage conditions, there was no indication of mass loss in any of the unexposed posts.

Metsä-Kortelainen and Viitanen (2017) tested thermally modified specimens in soil contact. They also reported severe decay in the bottom and middle specimens, while the uppermost specimens remained almost intact during the exposure of 6 years. Kleindienst *et al.* (2017) reported similar results for the soil contact test of specimens with dimensions of 500 mm × 25 mm × 25 mm. The moisture content is usually high at the ground level and bottom position, whereas the top part of the post may dry out even during wet periods (Blom and Bergström 2006).

3.2. Pilodyn indentation hardness

Figure 1 shows the Pilodyn indentation depths by post size, treatment, and exposure group. One must bear in mind that in the case of exposed posts, the untreated control specimens, indicated by the dashed reference line, represent only posts that were solid enough to be accepted in tests.

Based on the Pilodyn test, the circular posts were harder than the square-shaped ones in the case of CTO-treated specimens. The size or shape of the exposed control and copper salt-impregnated posts did not affect the Pilodyn indentation depth. On the other hand, square-shaped CTO I-impregnated posts were significantly softer ($p < 0.001$) than the round ones after the soil contact test. The size did not affect the Pilodyn indentation depth of the soil contact-tested square-shaped posts, whereas the 120 mm diameter round posts were significantly harder ($p = 0.011$) than the 80 mm posts after the nine-year soil contact. It is notable that in the non-exposed condition, i.e. intact wood, the average Pilodyn indentation depth in the circular posts was significantly lower ($p < 0.001$) than in the square-shaped posts. Either the shape itself or the processing method (peeling vs. sawing) affects the surface hardness determined by the Pilodyn method.

3.3. Basic density

The average basic densities of specimens by post size, treatment, exposure, and within-post position are presented in Figure 2. Unlike expected, basic density was not affected by the vertical position of the specimen in the post ($p = 0.866$). This may be related to the differences in amounts of preservative leached from the three specimen positions during the exposure period. In case of CTO I and CTO II posts, however, visually evaluated amount of leaching was not affected by the location of the specimen. In addition, tendency to leach was strongly correlated with the basic density: the higher density the more leaching. The basic density had a moderate negative correlation with the Pilodyn indentation depth ($r = -0.59$, $p < 0.001$, $N = 222$), which is in line with the findings by Greaves *et al.* (1996), Wu *et al.* (2011), and Couto *et al.* (2013).

Basic densities of exposed control specimens represent only a minority of all posts, since most of the control posts were excluded from analyses due to extensive softening caused by biodegradation. Still, a significant difference was observed between the basic densities of treated and control specimens ($p < 0.001$) after nine years of soil contact. Basic densities of CTO I- and CTO II-treated specimens were significantly higher than those of control ($p < 0.001$ for both) and copper salt-impregnated specimens ($p < 0.001$ for both). No differences were observed between CTO I and CTO II ($p = 0.729$), or between control and copper salt-impregnated specimens ($p = 0.386$). In case of non-exposed posts, the basic densities differed significantly between treated and control specimens ($p < 0.001$). There was no difference between control and copper salt-impregnated specimens ($p = 0.010$) or between CTO I- and CTO II-treated specimens ($p = 0.078$). Furthermore, the basic densities of exposed and non-exposed posts differed

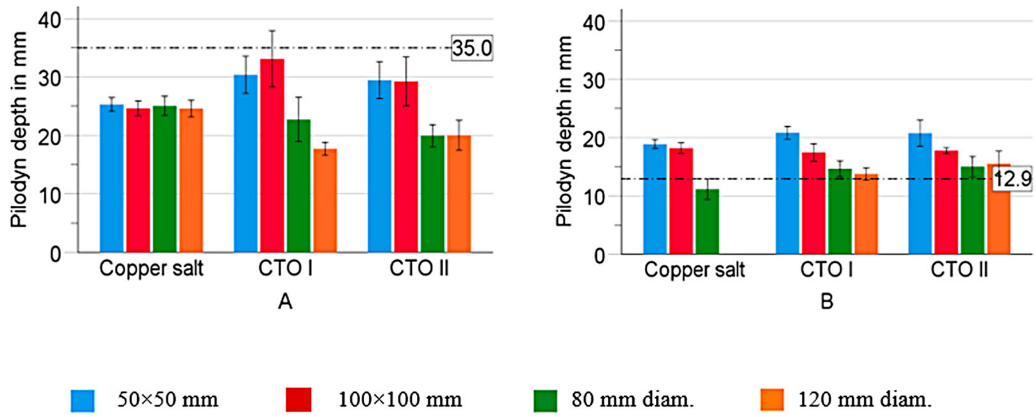

Figure 1. The Pilodyn indentation depths (mm) at the ground level according to the size, shape, and treatment of the post. Graph A represents the soil contact exposed posts and the dashed reference line indicates the average Pilodyn indentation depth for the non-treated control posts. Graph B represents the non-exposed posts that were stored indoors, and the reference line represents the average Pilodyn value for the untreated posts.

only between CTO I ($p = 0.001$) and CTO II ($p < 0.001$), whereas no difference was observed between exposed and non-exposed copper salt impregnated ($p = 0.237$) or control specimens ($p = 0.198$). In case of control and copper salt-treated group, the leaching of the extractives and treated chemical due to the weathering did not make a difference because extractives in wood typically represents less than ten per cent of its dry mass (e.g. Routa *et al*. 2017). For CTO I- and CTO II-treated specimens, a major contribution was possibly due to the leaching of the tall oil, which is caused by insufficient curing of oil inside wood, as reported by Hyvönen *et al*. (2007).

After the soil contact of nine years, the basic density of square-shaped copper salt-impregnated posts was higher than that of the round ones ($p < 0.001$). Once all specimens consisted solely of sapwood, there is no logical explanation for this finding. For the square-shaped posts, the average density of 100 × 100 mm specimens was higher than that of 50 × 50 mm specimens ($p = 0.036$). Hence, a larger proportion of the smaller posts was decayed during the exposure period. However, no differences were observed between the average densities of different-sized round posts. Basic densities of non-exposed posts did not differ between the post shapes or sizes within the same treatments.

For both CTO I and CTO II-impregnated specimens exposed to soil contact, the basic density of round specimens was significantly higher than that of square-shaped specimens ($p < 0.001$ for both), which was also observed for the non-exposed specimens ($p < 0.001$). Also here, the findings may reflect more intense CTO uptake in the round posts than in the square-shaped ones during the impregnation – also leaching was more intense in the round specimens. Size did not affect the density of either the square or round posts (Figure 3).

3.4. Deformation under static compression

Small compression deformation indicates high parallel-to-the-grain stiffness of the specimen, yet the modulus of elasticity, *per se*, was not determined. Again, only a fraction of untreated control specimens survived from the soil contact

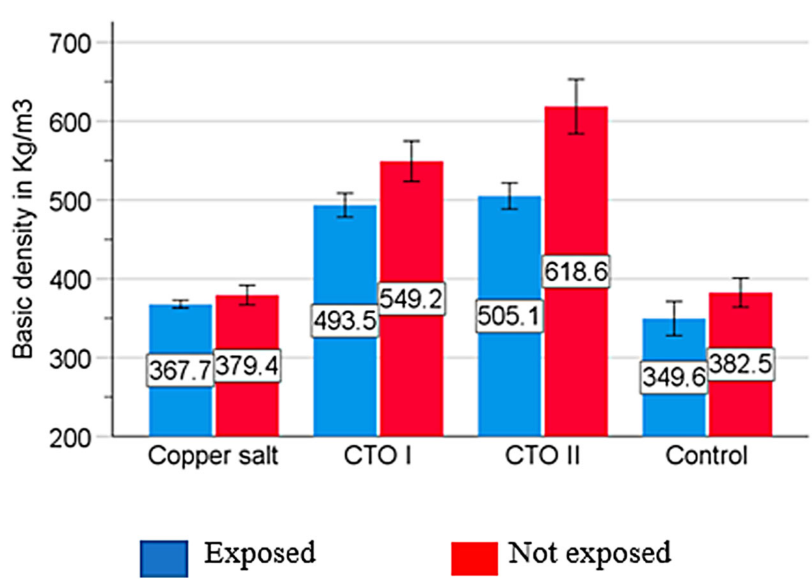

Figure 2. Average basic densities of exposed and non-exposed posts per treatment.

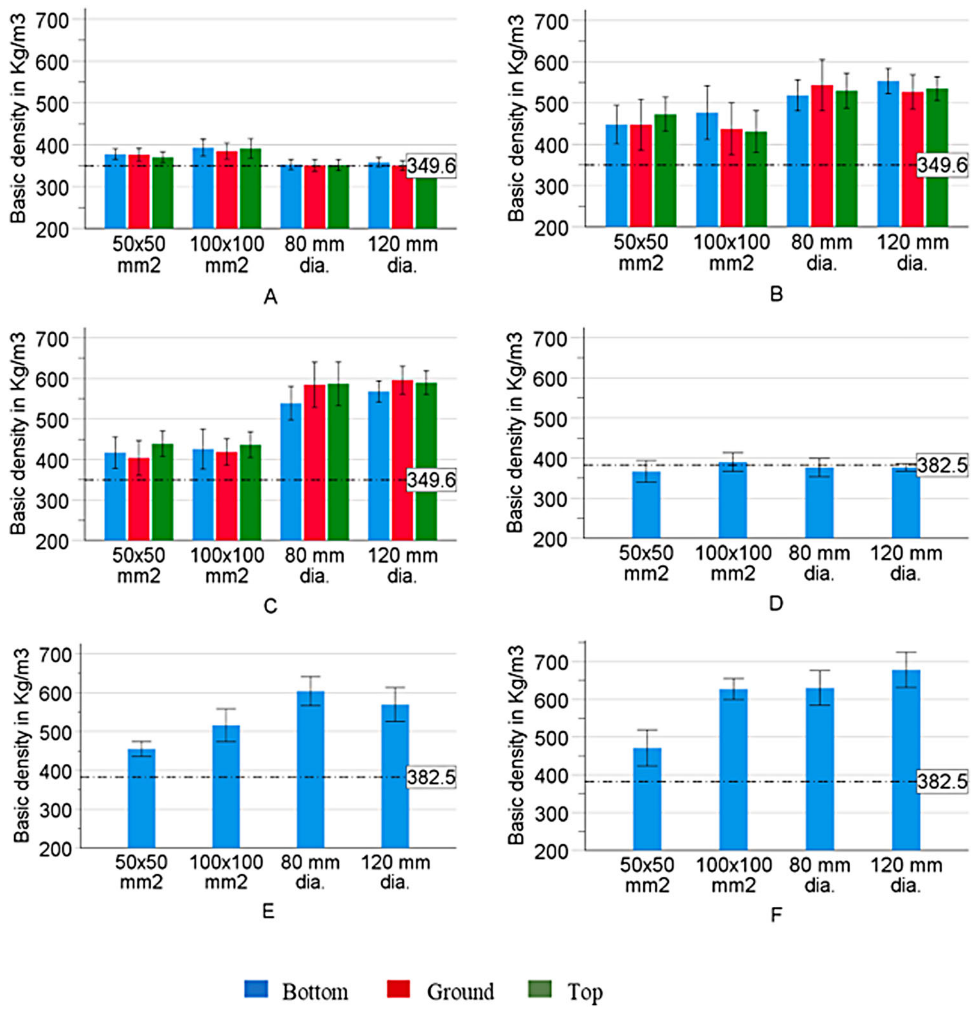

Figure 3. Average basic densities of specimens at different positions within the posts after nine years of soil contact according to the size and treatment. Graphs A, B and C represent the copper salt, CTO I- and CTO II-treated specimens from soil contact exposed posts, respectively, and the horizontal reference line stands for the average basic density of untreated control posts. Graphs D, E, and F represent the copper salt, CTO I- and CTO II-treated specimens from non-exposed posts, respectively, and the horizontal reference line stands for the average basic density of the untreated non-exposed posts.

of nine years in a condition allowing specimen preparation. Therefore, the control group results presented do not represent the average values of all controls but the average of the survivors.

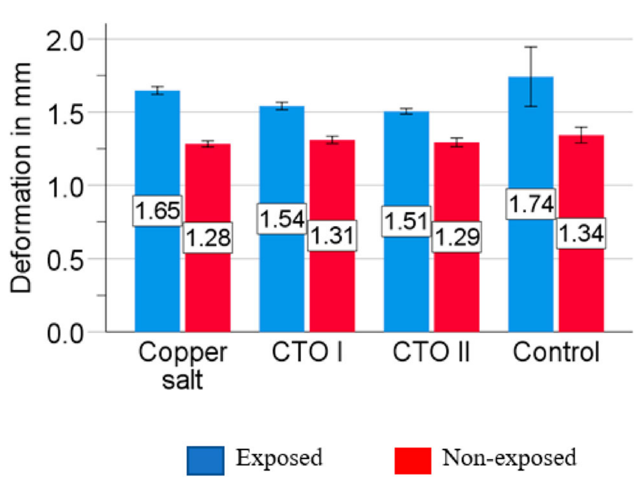

Figure 4. Deformation of specimens from different treatments under a static 45,000 N compression.

The average compression deformations under a static 45,000 N compression were smaller in the topmost soil contact exposed specimens in virtually all post shapes, sizes, and treatments than in the specimens from ground level or underground. Compression deformations differed significantly between the exposed treatment groups ($p < 0.001$) (Figure 4). The copper salt-impregnated specimens deformed significantly more than the CTO I ($p < 0.001$) and CTO II ($p < 0.001$) impregnated specimens. No differences were observed in the deformations between the non-exposed treatment groups ($p = 0.341$).

The compression deformation was positively correlated with the Pilodyn indentation depth ($r = 0.48$, $p < 0.001$, $N = 216$) and negatively correlated with the basic density ($r = -0.48$, $p < 0.001$, $N = 612$).

A significant difference between ground level and other positions was evident only for 50 × 50 mm copper salt-treated specimens, in which the ground level specimens deformed more than the specimens from other heights. The top specimens had the lowest deformation in most cases.

Kleindienst *et al*. (2017), who carried out soil contact tests with 500 mm × 25 mm × 25 mm sticks, also reported an increment of strength from the bottom to the top of the specimens.

Specimen size did not affect the compression deformation in any of the treatment, exposure, or shape groups except for the CTO I-treated specimens in the exposed condition, in which the 50 × 50 mm specimens deformed more than the larger ones. Smaller specimens may be affected more than bigger ones by biodegradation (Nicholas and Crawford 2003).

4. Conclusions

The copper salt (NTR AB class) and crude tall oil (CTO) impregnation caused a significant improvement against mass loss during the nine years of soil contact, though some mass loss was observed in the CTO-treated specimens, too. Only one-fifth of the untreated posts survived in a reasonable condition. One must keep in mind that the NTR AB class, which was used in this study, is usually recommended for above-ground uses, whereas the recommended soil contact or underground application impregnation class is NTR A. In terms of the specimen's vertical location within the post, the top specimens (above ground level) were less likely to decay, while the difference between the ground level and underground specimens was hardly observable.

After the exposure period, the Pilodyn indentation hardness of the treated posts was significantly higher than that of untreated control posts. However, differences among the treatment groups were not consistent. The circular posts indicated higher hardness, except for the exposed copper salt-impregnated post. The size effect was partly inconsistent, but generally, higher hardness was observed in larger posts. The CTO-treated posts had the highest basic density. The basic densities of specimens prepared from different heights were equal, indicating the minimum or no effect of the soil contact and exposure on the material density. The copper salt-treated specimens deformed significantly more than the CTO I- and CTO II-treated specimens, and unexpectedly, the untreated control specimens did not differ significantly from the treated ones.

Nine years of soil contact test indicates that CTO impregnation is a competitive and environmentally sound wood protection method in terms of mass loss, surface hardness, and mechanical performance. CTO's I and II performed similarly.

Our tests indicated that Pilodyn indentation hardness was not the best option for testing the hardness of the exposed posts because the indentation depth of severely softened specimens typically exceeds the measurement limit of 40 mm. A bigger diameter needle would have helped in those cases but would not have provided good results in harder specimens. We did not analyse the hidden knots that may have influenced at least the density and compression results. The probability of hidden knots is higher in larger specimens than in the small ones. Finally, the soil properties are crucial for the decay processes, and the experiments could be repeated in different places to assess the performance of treatments in a broader range of exposure conditions.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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