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# DECAY RESISTANCE, DIMENSIONAL STABILITY AND MECHANICAL STRENGTH OF POPLAR WOOD MODIFIED WITH PLANT-DERIVED COMPOUNDS

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## ABSTRACT

The cinnamaldehyde, salicylic acid, stearolic acid and citric acid were plant-derived organic compounds that can be activated to fungi, that could degrade the wood in long term. The compounds with concentrations of 3%, 5% and 7% assisted by different dispersants were impregnated into poplar (*Populus nigra L.*) specimens by the vacuum-pressure method. After that, weight percentage gain (WPG), decay resistance against white-rot fungi (*Trametes versicolor*) and brown-rot fungi (*Gloeophyllum trabeum*), color change, dimensional stability and mechanical properties including modulus of elasticity (MOE) and modulus of rupture (MOR) were measured. The results indicated that cinnamaldehyde impregnated poplar showed antifungi activity against both *G. trabeum* and *T. versicolor*, and citric acid impregnated poplar showed antifungi activity against *G. trabeum*. The color of poplar specimens before and after impregnated cinnamaldehyde and citric acid had a little change, dimensional stability had been improved and mechanical properties especially for MOR increased significantly.

KEYWORDS: Wood preservatives, natural organic compounds, decay resistance, dimensional stability, MOE, MOR.

## INTRODUCTION

Wood is a useful natural resource. It can be recycled and reused and plays a significant role in human activities (Teng et al. 2018). For applications, wood is expected to possess certain durability against fungal attacks, as well as a good dimensional stability (Coggins 2008, Ali et al. 2011, Aydin et al. 2015). The application of preservative chemicals is one of the most common wood preservation methods (Mubarok 2019).

The current inorganic preservatives pose the potential risks to the environment and human health (Borges et al. 2000, Khan et al. 2006, Nkansah et al. 2015, Brocco et al. 2017). The safer alternatives have been sought by many researchers. More and more researches on finding nuisance-free wood preservatives have been done. Many plants produced potent natural compounds which might be used as alternatives to protect wood or wood-based materials (Mohammed et al. 2016, Tchinda et al. 2018).

The plant-derived chemicals have low harmful environmental impact. For example, several plant-derived origin compounds have been found to be effective against wood-decaying fungi, such as essential oils (Kartal et al. 2006, Xie et al. 2017), tannins (Anttila et al. 2013, Tondi et al. 2015), flavonoids (Treutter 2006), alkaloids (Wang et al. 2012) and extracts of konjac flying powder (Bi et al. 2019) and coconut shell (Shiny 2018), Cameroonian woods (Tchinda et al. 2018) and eucalyptus (Gonzalez-Laredo et al. 2015).

The four natural organic compounds (cinnamaldehyde, salicylic acid, stearolic acid and citric acid) were selected. They are biodegradable and environment-friendly compared to traditional inorganic preservative. For example, cinnamaldehyde is major compound in the cinnamon essential oil and can be extracted from bark and leaves of cinnamon trees. Salicylic acid was found in willow bark and other plants as endogenous plant hormone (Asghari et al. 2010). Stearic acid can be extracted from palm oil. And citric acid is also distributed in nature widely in the citric trees. These four natural organic compounds have been used in food and cosmetics industries as preservative, or additive in pharmaceutical industry. For example, the cinnamaldehyde could be approved for apply a food flavoring and also had antimicrobial properties to extend food shelf-life (Nostro et al. 2012).

In this study, cinnamaldehyde, salicylic acid, stearolic acid and citric acid were chosen to treat poplar wood to increase the decay resistance of against white-rot fungi (*Trametes versicolor*) and brown-rot fungi (*Gloeophyllum trabeum*). The color change, dimensional stability and mechanical property of treated poplar specimens were recorded. The results of this study can provide the guidance for the natural organic plant-derived compounds as eco-friendly wood preservatives.

## MATERIALS AND METHODS

### Sample preparation

Poplar (*Populus nigra L.*) log with a height of 180 cm and a diameter of 20 cm at breast height was obtained from poplar plantations in Jiaozuo city, Henan Province, China. Wood specimens were cut into wood blocks. Six repetitions sized 20 mm (R) × 20 mm (T) × 10 mm (L) were chosen for detecting wood decay resistance study, six repetitions sized 20 mm (R) × 20 mm (T) × 20 mm (L) for detecting color and dimensional stability study and ten repetitions sized 20 mm (R) × 20 mm (T) × 300 mm (L) for detecting mechanical property study. The specimens were oven dried at 60°C until constant mass ($M_0$) which their moisture content reached 8%.

### Preparation of impregnated wood

The vacuum-pressure method was used to infuse the solution into wood, using capsule-shaped vacuum-pressure equipment. Dispersants and dissolution methods of cinnamaldehyde, salicylic acid, stearolic acid and citric acid were listed in Tab. 1. The mixed solutions are formulated into concentrations of 3%, 5% and 7% respectively to impregnate specimens under vacuum condition (-0.08 MPa) for 30 min, followed by 15-min pressure treatment (0.8 MPa). After treatment all specimens were kept in solution for 12 hours. The treated poplar specimens were then oven dried at 60°C until constant mass ($M_1$).

*Tab. 1: Dispersants and dissolution methods for natural organic compounds.*

| Organic compounds | Dispersant | Solvent | Dissolving temperature | Dissolving method |
|---|---|---|---|---|
| Cinnamaldehyde | Cetyltrimethylammonium bromide | Water | Room temperature | Ultrasonic processing |
| Salicylic acid | Sodium lignosulfonate | Water | Room temperature | Ultrasonic processing |
| Stearolic acid | | 70% alcohol | 60°C | Heat dissolved |
| Citric acid | | Water | Room temperature | Ultrasonic processing |

**Weight percentage gain (WPG) of poplar samples after impregnation**

The WPG was calculated based on the variations in the weight before and after impregnation according to Eq. 1:

$$WPG = (M_1 - M_0) / M_0 \times 100\% \qquad (1)$$

where: $M_0$ - constant mass of poplar specimen before impregnation, (g)
$M_1$ - constant mass of poplar specimen after impregnation, (g).

**Decay resistance determination**

Decay resistance of impregnated poplar specimens was evaluated after the treated samples exposed to the white-rot fungus (*Trametes versicolor*) or the brown-rot fungus (*Gloeophyllum trabeum*). The culture bottles were filled with 150 g river sand, 75 g saw-dust, 4.3 g corn flour, 0.5 g brown sugar. 9.4 g maltose was dissolved in 100ml water and it was poured into the culture bottles slowly. Two poplar (*Populus tomentosa*) feeder blocks with dimensions of 22 mm (R) × 22 mm (T) × 3 mm (L) were placed into culture bottles. Culture bottles were then sterilized at 121°C for 1 hour, after prepared. The fungi having grown on potato dextrose agars for 7 days were inoculated on culture bottles. The culture bottles were incubated in darkness at 28°C and 80% relative humidity for 7 days until the feeder blocks full with fungal mycelium.

Impregnated poplar specimens were sterilized at 105°C for 30 min and placed onto feeder blocks in culture bottles. Each culture bottle contained two specimens. Then culture bottles with impregnated poplar specimens were incubated at 28°C and 80% relative humidity for 12 weeks. Six replicates with dimensions of 20 mm (R) × 20 mm (T) × 10 mm (L) were done in each group. After incubation in 12 weeks period, poplar specimens were taken out. The mycelium of fungi wiped off and the samples were dried at 60°C to obtain constant mass (M2). The mass loss (ML) caused by fungi was calculated using Eq. 2, and the decay resistance was determined according to the ASTM rating (ASTM, 2005) (Tab. 2).

$$ML = (M_1 - M_2) / M_1 \times 100\% \qquad (2)$$

where: ML - mass loss after decay test, (%)
$M_1$ - mass of specimen the after impregnation, (g)
$M_2$ - mass of specimen after decay test, (g).

*Tab. 2: Natural decay resistance rating standard of wood established by the ASTM D2017 standard.*

| Class of decay resistance | Mass loss (%) |
|---|---|
| Highly resistant | 0 - 10 |
| Resistant | 11 - 24 |
| Moderately resistant | 25 - 44 |
| No resistant | ≥ 45 |

### Scanning electron microscopy of decayed wood

A scanning electronic microscope (SEM, Hitachi S-4800, Japan) was used to observe mycelial distribution and the degradation of wood structure caused by fungi. The microscope was operated at acceleration voltage of 20 kV. Small samples sized 10 × 10 × 1 $mm^3$ were cut from the untreated poplar and decayed poplar and coated with a layer of platinum about 20 nm thick before observation. SEM images of poplar samples impregnated with concentrations of 7% natural organic compounds were chosen in the SEM study.

### Color measurement

The surface color measurements of poplar specimens before and after impregnation were recorded with Spectrophotometer CS-820. The color of cross directions was measured. Six clear wood samples without any defects were used as replicates in each concentration of each compound. The L* describes the lightness, a* and b* describe the chromatic coordinates on the green-red and blue-yellow axis, resp. From the L*a*b* values, the difference in the lightness (ΔL*) and chroma coordinates (Δa* and Δb*), and total color difference (ΔE) were calculated using the following Eq. 3 (Bekhta et al. 2003):

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \qquad (3)$$
$$\Delta L^* = Li^* - L0^*$$
$$\Delta a^* = ai^* - a0^*$$
$$\Delta b^* = bi^* - b0^*$$

where: ΔE* - the total color difference, ΔL* - the lightness difference before and after impregnation, Δa*- the difference of chromatic coordinates on the green-red axis before and after impregnation, Δb* - the difference of chromatic coordinates on the blue-yellow axis before and after impregnation.

### Determination of wood dimensional stability

Poplar specimens with dimensions of 20 mm (R) × 20 mm (T) × 20 mm (L) were divided into treated groups and control groups. Six replicates were chosen in each concentration of each compound. Treated groups and control groups were oven dried at 60°C until a constant weight. The dimensions of three sections (radial, tangential and cross) were measured and volumes were calculated ($V_0$). Poplar specimens with a constant weight were respectively stored in a temperature and humidity controlled incubator at 30°C and 65%, 70%, 80%, 85%, 80%, 70%, 65% relative humidity to reach the equilibrium moisture content (EMC). Dimensions of three sections and calculate volumes at above-mentioned different relative humidity were measured (Vw). Volume increase rate was calculated according to Eq. 4:

$$\Delta V = (V_w - V_0) / V_0 \times 100\% \quad (4)$$

where: $V_0$ - the initial volume of the specimen, ($mm^3$)
$V_w$ - the volume of the specimen after conditioning, ($mm^3$).

**Mechanical property tests**

According to ASTM Standard D143, impregnated poplar specimens with dimensions of 20 mm (R) × 20 mm (T) × 300 mm (L) were tested to failure in third point loading by using a loading speed of 3 $mm \cdot min^{-1}$. Distance between two supports was set to 240 mm. And ten clear poplar specimens were chosen as replicates. Treated groups were impregnated through the above step and oven-dried at 60°C which their moisture content reached 8%. Modulus of elasticity (MOE) and modulus of rupture (MOR) of impregnated specimens and blank control groups were determined using a universal testing machine. Experimental results were statistically analyzed by SPSS Version 17 software (IBM Corp., Armonk, NY, USA). Two-way analysis of variance (ANOVA) and were applied to determine the significant differences at a significance level of $p < 0.05$.

## RESULTS AND DISCUSSION

**Weight percentage gain (WPG)**

WPG of the impregnated poplar specimens were was presented in Fig. 1.

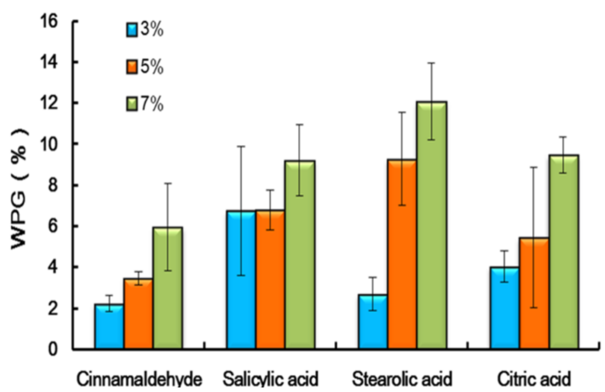


*Fig. 1: WPG of the specimens which were impregnated with different concentration of natural organic compounds.*

The specimens impregnated with 7% concentration stearolic acid have maximum weight increasing, with their WPG about 12.08%. Specimens impregnated with 3% cinnamaldehyde have the lowest weight gain, and their WPGs were 2.24%. Generally, as the concentration of the compounds raised, the WPGs of the specimens also increased.

**Decay resistance and SEM observation of the impregnated poplar**

The mass losses and decay rating of impregnated poplar wood attached by *G. trabeum* and *T. versicolor* were presented in Tab. 3. Mass losses in the control group reached 31.93% and 55.98% against *G. trabeum* and *T. versicolor.* These losses would cause considerable reductions in wood properties (Wilcox 1978). Mass losses of poplar specimens impregnated with concentration of 5%, 7% cinnamaldehyde under the culture conditions for *T. versicolor* reached 5.02% and 1.63% respectively. Excellent decay resistances of cinnamaldehyde against both *G. trabeum* and *T. versicolor* were showed, the results were consistent with Yang et al. (2017) who reported that decay resistance rating reached highly resistant for *Populus ussuriensis* Kom. impregnated with 50 $mg \cdot ml^{-1}$ cinnamaldehyde.

At the concentrations of 5% and 7% cinnamaldehyde, the treated wood against *G. trabeum* reached the high decay resistance rating. And mass losses of poplar specimens impregnated with concentration of 3%, 5% and 7% cinnamaldehyde against *G. trabeum* were less than 3%. For citric acid, the effects of decay resistances for *T. versicolor* were not very effective. However, poplar specimens impregnated with citric acid had better decay resistances against *G. trabeum*. And the effects of decay resistance treated by cinnamaldehyde and citric acid increased as impregnation concentration increased. The effect of salicylic acid and stearolic acid on the growth of *G. trabeum* and *T. versicolor* were not significant. And their antifungal activity was much weaker than that of cinnamaldehyde or citric acid.

For salicylic acid, it showed antifungi activity against both *G. trabeum* and *T. versicolor* in five-week decay resistant research reported by Bi et al. (2019), however it did not inhibit or even promote the growth of fungi. The results of this experimental phenomenon need further tests to better understand antifungi mechanism for salicylic acid as wood preservative. The mass losses of stearolic acid impregnated poplar specimens might reflect solubility issues.

The SEM images of poplar specimens impregnated with 7% concentration after 12 weeks of exposure to *G. trabeum* and *T. versicolor* were presented in Fig. 2. From blank control samples as well as samples treated by 7% salicylic acid and stearolic acid, severe damage to the poplar cell wall occurred, which were consistent with Cai et al. (2020). Mycelia were full in shape. However, mycelia were slim and were inhibited in Fig. 2d and Fig. 2j (marked with circles). And the cell wall was practically intact (marked by a rectangle). The cinnamaldehyde can inhibit the growth of for *G. trabeum* and *T. versicolor,* and the citric acid can inhibit the growth of *G. trabeum* through the SEM observation.

*Tab. 2: Natural decay resistance rating standard of wood established by the ASTM D2017 standard after exposed to G. trabeum or T. versicolor.*

| Fungi | Types of preservatives | Concentration | Mass loss (%) | Rating |
|---|---|---|---|---|
| *G. trabeum* | Blank control | | 31.93 (7.42) | |
| | Cinnamaldehyde | 3% | 2.84 (2.43) | Highly resistant |

| | | | | |
|---|---|---|---|---|
| | | 5% | 2.32 (0.48) | Highly resistant |
| | | 7% | 0.21 (3.15) | Highly resistant |
| | Salicylic acid | 3% | 37.04 (3.06) | Moderately resistant |
| | | 5% | 40.59 (4.73) | Moderately resistant |
| | | 7% | 47.67 (4.09) | No resistant |
| | Stearolic acid | 3% | 40.82 (3.31) | Moderately resistant |
| | | 5% | 41.77 (4.36) | Moderately resistant |
| | | 7% | 40.37 (6.39) | Moderately resistant |
| | Citric acid | 3% | 24.31 (3.20) | Moderately resistant |
| | | 5% | 17.87 (5.04) | Resistant |
| | | 7% | 10.53 (5.29) | Resistant |
| *T. versicolor* | Blank control | | 55.99 (4.49) | |
| | Cinnamaldehyde | 3% | 22.68 (10.92) | Resistant |
| | | 5% | 5.02 (2.20) | Highly resistant |
| | | 7% | 1.63 (2.02) | Highly resistant |
| | Salicylic acid | 3% | 41.36 (8.55) | Moderately resistant |
| | | 5% | 48.97 (5.17) | No resistant |
| | | 7% | 45.74 (6.83) | No resistant |
| | Stearolic acid | 3% | 50.46 (4.02) | No resistant |
| | | 5% | 46.37 (2.56) | No resistant |
| | | 7% | 50.28 (7.09) | No resistant |
| | Citric acid | 3% | 65.41 (5.53) | No resistant |
| | | 5% | 66.38 (4.05) | No resistant |
| | | 7% | 57.34 (6.08) | No resistant |

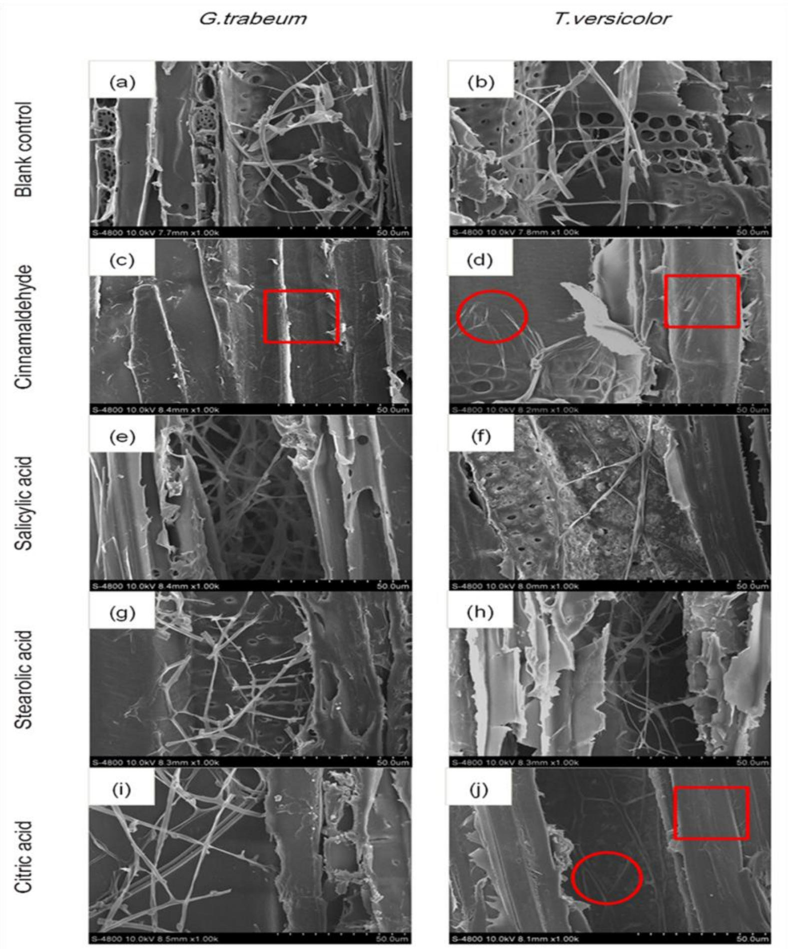


*Fig. 2: SEM images of poplar samples impregnated with 7% natural organic compounds after the decay test.*

## Wood color change of after impregnation

The color change of poplar specimens before and after they were impregnated with various compounds were presented in Fig. 3. The L *, a *, b * and ΔE* were showed in Fig. 4. The L*, a *and b * parameters did not change much after treatment. ΔE* were remained within 10%. L* parameters of poplar samples impregnated with salicylic acid was less than that of blank control groups and other treated groups and showed darkness in wood. The color changed more as the concentration of the compounds increased.

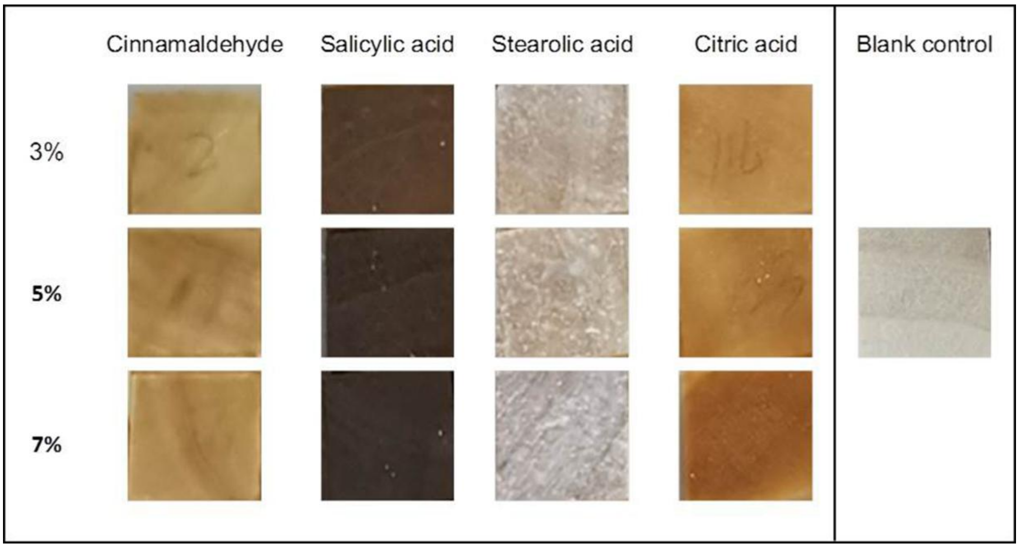


*Fig. 3: The color of poplar specimens after impregnated with different compounds.*

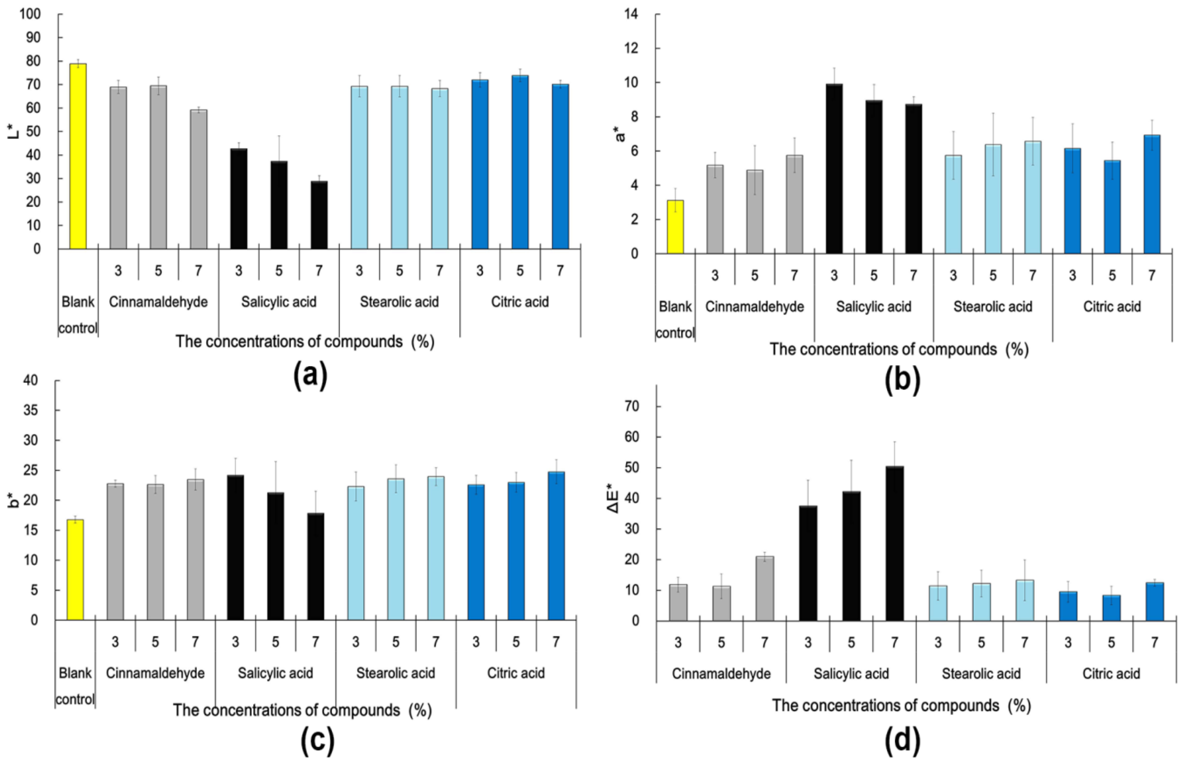


*Fig. 4: L *, a *, b * and ΔE* in poplar specimens changed after impregnated with different compounds.*

**Dimensional stability of the impregnated poplar**

Volume change of impregnated poplar specimens with cinnamaldehyde, salicylic acid, stearolic acid and citric acid, respectively and blank control groups were presented in Fig. 5. Volumes of specimens increased most when they were stored in a 30°C and 85% relative humidity (RH) compared to other temperature and RH. Volume increase of untreated poplar specimens reached 6.13%. Volume increase of poplar specimens impregnated with cinnamaldehyde and citric acid had a significant reduction compared to the blank control group, close to 2%. And concentration of impregnated cinnamaldehyde and citric acid had little effect on reduction of volume increase rates in poplar specimens. This result showed that wood impregnated with cinnamaldehyde or citric acid has better dimensional stability.

Natural organic compounds might be impregnated into vessel and they would block the passages of water transport (He 2019), thus dimensional stability of impregnated poplar specimens were improved.

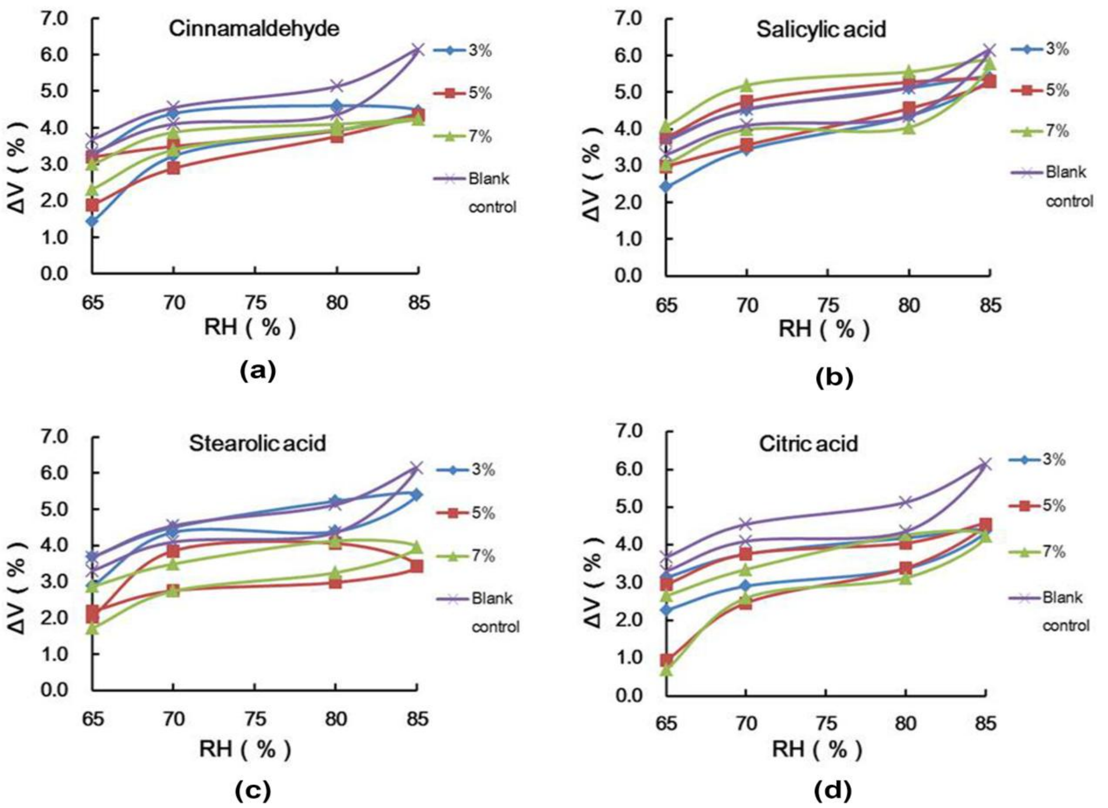


*Fig. 5: Volume increase (ΔV) of poplar specimens after impregnated with different compounds.*

**Mechanical properties after impregnation**

Mechanical properties, including modulus of elasticity (MOE) and modulus of rupture (MOR) after impregnation was presented in Fig. 6. For MOE (Fig. 6a), blank control groups reached 6291 MPa. Compounds type and compounds concentration had no significant effects on MOE ($p = 0.08$ and 0.06, resp.). MOE of other poplar specimens impregnated with four natural organic compounds was improved differently, and MOE of 3% cinnamaldehyde impregnated poplar specimens decreased slightly. MOEs of impregnated specimens were positive correlation with concentration of cinnamaldehyde, salicylic acid, stearolic, however, citric acid group had opposite result. MOR of 7% cinnamaldehyde and 7% salicylic acid impregnated specimens reached 7339 MPa and 8241 MPa, improving 16% and 30% respectively compared to blank control groups.

For MOR (Fig. 6b), blank control groups reached 89 MPa. Compounds type and compounds concentration had significant effects on MOR (p-value = 0.006 and 0.018 resp.). MORs of treated groups all improved compared to blank control groups. MORs of impregnated specimens were positive correlation with concentration of impregnated compounds. Cinnamaldehyde and salicylic acid impregnated specimens promoted the highest among four kinds of natural organic compounds, and MOR of 7% cinnamaldehyde and 7% salicylic acid impregnated specimens reached 107 MPa and 114 MPa, improving 20% and 28% respectively compared to blank control groups.

MOR and MOE of four kinds of natural organic compounds were improved maybe it was because compounds get into the cell and strengthened the cell wall, it was consistent with the research of Bian et al. (2019).

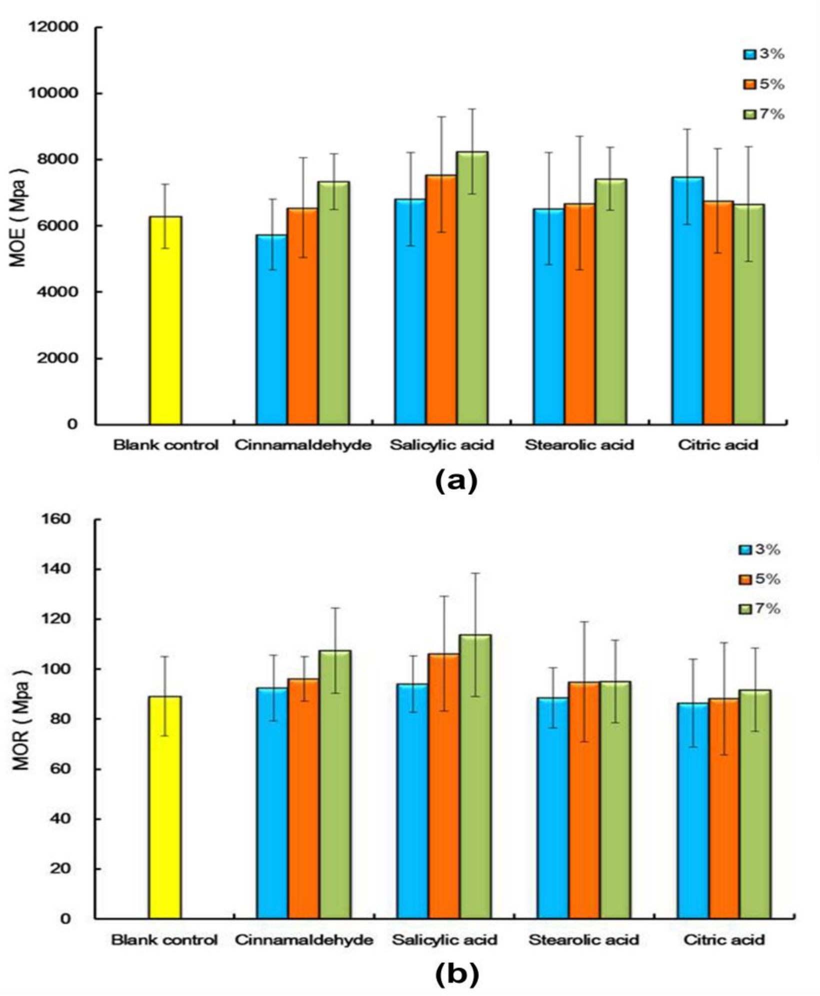


*Fig. 6: Mechanical properties of poplar specimens after impregnated with different compounds: (a) MOE, (b) MOR.*

## CONCLUSIONS

Cinnamaldehyde, salicylic acid, stearolic acid and citric acid as active substances were impregnated into poplar wood using the vacuum-pressure method. WPG of impregnated specimens improved as the concentration increased. Wood impregnated with cinnamaldehyde showed the antifungi activity against both *G. trabeum* and *T. versicolor*, and impregnated poplar with citric acid showed the antifungi activity against *G. trabeum*. The impregnation of cinnamaldehyde, stearolic acid and citric acid did not cause large color change. Cinnamaldehyde and citric acid decreased volume increase rates thus improved dimensional stability. Mechanical properties of impregnated specimens had been improved, and compounds type and compounds concentration had no significant effects on MOE, however, they had significant effects on MOR.

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