

IMPROVING THE COLOR STABILITY OF WOOD-PLASTIC COMPOSITES THROUGH FIBER PRE-TREATMENT¹

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Abstract. Wood-plastic composites (WPCs) continue to make inroads into the decking market. One of the main drawbacks from the consumer's perspective is that they can fade during weathering. It has been shown that WPCs fade more when exposed to a combination of light and water spray than when exposed only to light. This suggests that the loss of the color-imparting extractives from the wood could be partly to blame for color fade. Pigments are routinely added to only the plastic matrix to combat WPC color fade despite the fact that change in color could be due primarily to changes in the wood component of WPCs. In this study, we investigated two methods of fiber pretreatment in an attempt to improve the color stability of WPCs. The first was to remove water-soluble extractives from wood flour through a series of washes. Washed wood flour was then compounded with high-density polyethylene and injection-molded into test specimens. The second method involved coloring wood flour with either a water-based dye or an oil-based stain. Colored wood flour was also compounded with high-density polyethylene and injection-molded into test specimens. Pigments were added to half of the composites containing colored wood. The composites underwent accelerated weathering in a xenon-arc weathering apparatus, and were monitored for changes in color. Washing wood flour and coloring wood flour with a water-based dye were ineffective for improving composite color stability during weathering. However, coloring wood flour with an oil-based stain resulted in a more color stable WPC.

Keywords: Wood-plastic composites, weathering, color, extractives, pigments.

INTRODUCTION

Wood-plastic composites (WPCs) have found their way into consumer, automotive, and construction applications, but are experiencing the fastest growth in exterior residential construction applications. WPC lumber is promoted as a low-maintenance alternative to solid wood. WPCs are often seen as "green" composites (ie

more natural, less toxic, etc.). Wood is a renewable component often sourced from a waste stream, and the entire composite can be recyclable (Clemons 2002). The introduction of WPCs in the decking market is mainly responsible for the fast growth, and WPCs are predicted to have 25% of the market by 2009 (Rossi 2005). A direct result of success in the decking market is that products are being developed and introduced for new exterior applications such as railings, fencing, roofing, and siding.

The growth in exterior applications results in a need to understand the weathering performance of WPCs, and several research groups have been working on characterizing and understanding changes that occur when WPCs weather (Bajwa and Bruce 2005; Colom et al 2000; Falk et al

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2000; Lundin 2001; Matuana et al 2001; Matuana and Kamdem 2002; Muasher and Sain 2006; Seldén et al 2004; Stark and Matuana 2003; Stark et al 2004; Stark and Matuana 2004; Stark and Matuana 2004a; Stark 2006; Stark and Matuana 2006). Of particular interest to the decking industry is how the color of WPCs changes after outdoor exposure. WPCs have been shown to lighten in color during weathering (Bajwa and Bruce 2005; Falk et al 2000; Lundin 2001; Matuana et al 2001; Muasher and Sain 2006; Stark and Matuana 2003; Stark et al 2004; Stark and Matuana 2004a; Stark 2006; Stark and Matuana 2006). Rowell et al (2000) reported the presence of a white chalky layer on the surface of an aspen fiber/polypropylene composite after weathering. This layer was easily scraped off and was used to show changes in material weight and thickness. The amount or rate of lightening can depend on manufacturing variables, weathering variables, and photostabilizers added.

Manufacturing method directly influences surface characteristics of WPCs and their response to weathering. WPCs with similar formulations manufactured differently, using injection molding or extrusion, experienced different rates of change in lightness (Stark et al 2004). Injection-molded composites lightened slower during weathering than extruded composites, largely due to the hydrophobic character of a plastic rich layer that formed on the surface of the composite during injection molding (Stark et al 2004).

The presence of moisture, when combined with UV exposure, also contributes to WPC lightening during weathering. When wood-flour-filled high-density polyethylene (HDPE) composites were exposed to xenon-arc radiation either with or without water spray in an accelerated weathering apparatus, more lightening of the composite color occurred after exposure to both xenon-arc radiation and water spray compared with exposure to xenon-arc radiation only (Stark 2006). UV radiation alone can lighten WPCs by causing the surface of the polymer matrix to crack, creating a whitening effect, and to a larger extent, by bleaching wood particles. Although UV

radiation contributes to wood particle bleaching, UV radiation with water exposure is deleterious for several reasons. However, the removal of water-soluble extractives was probably the main reason for the majority of color fade (Stark 2006).

One common method to improve the color retention of WPCs during weathering is to add photostabilizers. Photostabilizers that were developed for use in unfilled plastics are being adapted for use in WPCs. Pigments, light stabilizers, and UV absorbers (UVAs) have been investigated to improve the color stability of WPCs (Falk et al 2000; Lundin 2001; Matuana et al 2001; Muasher and Sain 2006; Stark and Matuana 2003; Stark and Matuana 2006).

Red and black pigments were shown to mitigate the increase in lightness of both polyethylene and polypropylene-based WPCs after accelerated weathering (Falk et al 2000). Titanium dioxide was shown to be an effective pigment for mitigating color changes in polyvinyl chloride-based WPCs (Matuana et al 2001). Lundin investigated the effect of hindered amine light stabilizer (HALS) content on the lightness and mechanical property loss of WPCs. The author reported that the addition of HALS to the composites did not affect color change caused by accelerated weathering (Lundin 2001). Stark and Matuana examined the effect of a low molecular weight HALS, a high molecular weight HALS, a benzotriazole UV absorber (UVA), and a pigment on the changes in lightness and mechanical properties of WPCs after weathering. Only the UVA and pigment significantly reduced composite lightening (Stark and Matuana 2003). Muasher and Sain (2006) also evaluated the performance of HALS and UVAs in stabilizing the color WPCs. They found that after weathering WPCs, high molecular weight diester HALS reduced some lightening. Also, adding a benzotriazole UVA to the HALS-stabilized WPC further improved color stability. By changing the concentrations of a UVA and pigment, we were able to conclude that the pigment was more effective at preventing lightening than the UVA (Stark and Matuana 2006). Bajwa and Bruce

(2005) reported smaller color shifts in WPCs after accelerated and natural weathering when concentrated color pigment was added, but larger shifts when mixed metal oxide pigment was added. Precolored wood-flour-filled WPCs were also evaluated, and resulted in a slightly smaller color shift compared with natural wood-flour-filled WPCs.

The wood component in WPCs is largely responsible for color fade. The color-imparting water-soluble extractives of the wood are likely removed during weathering, and may contribute to color fade. Adding a pigment to the plastic matrix has been shown to be an effective technique for reducing WPC color fade by camouflaging the bleaching wood particle, but coloring the wood component directly may be more effective. This study explores two different approaches to pretreat wood flour to improve WPC color stability during weathering: 1) wash the water-soluble extractives from the wood flour before manufacturing WPCs, and 2) stain or dye the wood flour before manufacturing WPCs. Wood-flour-filled HDPE composites containing pretreated wood flour were manufactured via injection molding, weathered, and analyzed for changes in color.

EXPERIMENTAL METHODS

Washed wood flour

Mixed-pine wood flour (PWF) was supplied by American Wood Fibers (AWF 4020; Schofield, WI). Salt cedar (*Tamarix chiensis*) wood flour (SCWF) was supplied as thinnings from the Bureau of Land Management. The thinnings were harvested from the Lower Colorado River Basin, and consisted of small-diameter logs approximately 7.6–15.2 cm in diameter. The logs were chipped, and then hammermilled through a 0.8-mm screen, and then hammermilled through a 0.8-mm screen. Both PWF and SCWF were screened through a 40-mesh screen (0.425 mm) to remove the larger particles. Fines were also removed; particles that passed through an 80-mesh screen (0.180 mm) were discarded.

To wash the wood flour, 0.23 kg of wood flour was combined with 2.5 L of tap water and heated to 80–85°C for 0.5 h while being continuously stirred. Water was then drained and the process repeated. After each subsequent washing, a sample of wood flour was removed for further analysis using a Soxhlet extraction in R.O. water for 3.5 h to allow us to determine the amount of remaining water-soluble extractives left in the wood flour after each washing. Both PWF and SCWF were washed 5 times, then oven-dried at 105°C. Unwashed and washed PWF and SCWF were incorporated into an HDPE matrix at both 25 and 50% by weight. The formulations studied are shown in Table 1.

Colored wood flour

PWF supplied by American Wood Fibers (AWF 4020) was uncolored; some of it was used as received, while color was added to other batches. Two materials were purchased to color the wood fiber, an oil-based stain (Semitransparent alkyd/oil stain, Clove Brown, Pittsburgh Paints, Pittsburgh, PA) and a water-based dye (TRANSFAST wood dye powder, Dark Mission Brown, Homestead Finishing Products, Cleveland, OH).

To color using oil-based stain, 0.45 kg of PWF was incorporated into 0.43 kg of stain, and stirred for 5 min. The stain was fully absorbed by the dry wood flour and required no draining. Stained wood flour (PWF-S) was dried at 60°C

TABLE 1. Wood-plastic composite formulations used to study the effect of washing wood flour on composite color stability.

Wood-flour type	Wood flour ^a (%)	HDPE ^a (%)
PWF	25	75
PWF–Washed	25	75
SCWF	25	75
SCWF–Washed	25	75
PWF	50	50
PWF–Washed	50	50
SCWF	50	50
SCWF–Washed	50	50

PWF = pine wood-flour, SCWF = salt cedar wood-flour

a: content based on weight percentage

for 24 h to remove volatiles. The net yield was 0.62 kg of PWF-S.

The dye was mixed per the manufacturer's recommendation (0.28 g dye in 1.9 L hot tap water). To dye the wood flour, 0.45 kg of PWF was added to the dye, and the mixture was stirred for 10 min. Excess dye was drained off (nearly fully absorbed), and the dyed wood flour (PWF-D) was dried at 105°C for 24 h. The net yield was 0.47 kg of PWF-D.

PWF, PWF-S, or PWF-D was incorporated into the composite at 50% by weight. To color the HDPE, a brown pigment supplied by Holland Colors Americas (Holcobatch Brown 936755; Richmond, IN) was incorporated into some of the composites at 2% by weight. The formulations studied are shown in Table 2.

Processing

Wood flour was compounded with HDPE (ExxonMobil HD 6605, 5 mfi, 948 kg/m³, Houston, TX) in a 1-L high-intensity thermokinetic mixer (K-Mixer, Synergistics, Inc, St. Remi de Napierville, Quebec) in 120-g batch sizes, run at 5500 rpm. An IR sensor monitored temperature, and the discharge temperature was set at 196°C for all the composites except those containing stained wood flour, where the discharge temperature was set at 177°C. The discharged, molten composite was pressed into a patty and allowed to cool at room temperature. The patty was granulated using a Ball and Jewell granulator outfitted with a 7.9-mm screen (Sterling, North Uxbridge, MA).

TABLE 2. Wood-plastic composite formulations used to study the effect of coloring wood flour on composite color stability.

Wood-flour type	Wood flour ^a (%)	HDPE ^a (%)	Pigment ^a (%)
PWF	50	50	0
PWF	50	48	2
PWF-S	50	50	0
PWF-S	50	48	2
PWF-D	50	50	0
PWF-D	50	48	2

PWF = pine wood-flour, S = stained, D = dyed

a: content based on weight percentage

Granulates were dried before being injection-molded. Composites were injection-molded into flexural specimens using a 33-tonne Cincinnati Milacron (Batavia, OH) injection molder. The mold nozzle temperature was 191°C, and mold temperature was 99°C. The ASTM mold cavity used for the flexural specimens was 127 × 3.2 × 12.7 mm (ASTM 2004).

Weathering

Composites were placed in a xenon-arc light exposure apparatus operated according to ASTM D 2565 (ASTM 2004a). Specimens were mounted in four rows on a drum that rotated around a xenon-arc bulb at 1 rpm. Each 2-h weathering cycle consisted of 108 min of xenon-arc radiation and 12 min of simultaneous xenon-arc radiation and UV exposure (ASTM 2004a). An irradiance sensor was used to measure light intensity for wavelengths from 300–400 nm. Irradiance was monitored, and voltage to the bulb was changed periodically to maintain constant irradiance. The radiant energy, or amount, of light energy to which specimens were subjected was calculated (irradiance × time). The specimens were harvested for analysis after 1000, 2000, and 3000 h of weathering, corresponding with a total radiant energy of 148, 300, and 450 MJ/m² at 300–400 nm, respectively.

Testing

A Minolta CR-200 Chroma Meter (Minolta Corporation, Ramsey, NJ) was used to measure color using the CIE 1976 L*a*b* color system (Robertson 1977). CIE L*a*b* is a three-dimensional color space measuring the lightness of the sample (L*) and color coordinates (a* and b*). L* ranges between 0 and 100 (black and white, respectively). An increase in L* means the sample is lightening. The color coordinates a* and b* range from –150 to +150. They are defined as the red/green coordinate, a* (+Δa* signifies a color shift toward red, –Δa* toward green) and the yellow/blue coordinate, b* (+Δb* toward yellow, –Δb* toward blue). Color was

measured in 2 locations of 5 replicate specimens.

Statistics

The mean and standard deviation of L^* for each formulation before and after weathering were calculated. Plots show changes in L^* ; each bar represents the mean of that data set and the error bars represent one standard deviation (Figs 2–4).

To determine significant differences between the means of L^* before and after 3000 h of weathering, Student's two-tailed t -tests, assuming unequal variance, were carried out at $\alpha = 0.05$. When reporting the L^* , superscript letters were used to denote significance (Tables 4–5). If the letters are the same, the hypothesis that the means were the same was accepted. In other words, the two means are not significantly different. The converse is that if the letters are different, the means are significantly different.

RESULTS AND DISCUSSION

Washed wood flour

Washing wood flour to remove water-soluble extractives was the first method evaluated to improve the color stability of WPCs. Wood flour (PWF and SCWF) was washed in a series of batch washes using a heated water bath. This simple method was chosen because it may be more adaptable by industry than other extractive removal methods. PWF-filled HDPE composites were evaluated because they are a typical WPC combination used in the decking market. In addition, SCWF-filled HDPE composites were evaluated because SCWF has a high water-soluble extractive content (Clemons and Stark 2007). It was visually verified that washing removed some extractives. It was evident that the bath water was colored after each wash and that the bath water became clearer after each subsequent washing. However, no chemical analysis of the water was performed.

The initial and remaining water-soluble extractive content of the wood flour after each washing was determined using Soxhlet extraction (Fig 1). The initial water-soluble extractive content of the PWF and SCWF was approximately 6 and 12%, respectively. After 5 washes, the remaining water-soluble extractive content for both PWF and SCWF was 3%. Therefore this simple washing method removed approximately 50% of the water-soluble extractives from PWF and 75% from SCWF. It is likely that there were other components removed from the SCWF during washing that were not strictly organic extractives. Salt cedar has a high salt content, and this would also be removed during washing (Clemons and Stark 2007).

The effect of washing on wood-flour color is reported in Table 3. Washing resulted in a slight decrease in lightness, L^* , for both PWF and SCWF. The red-green color coordinate, a^* , increased slightly for PWF and decreased for SCWF. After washing, the color of the PWF shifted slightly toward red, while the color of the SCWF shifted away from red. The yellow-blue color coordinate, b^* , decreased after washing for both PWF and SCWF. The color of both wood flours shifted slightly away from yellow after washing. Although washing caused color shifts in the wood flour, it did not cause the wood flour to lighten.

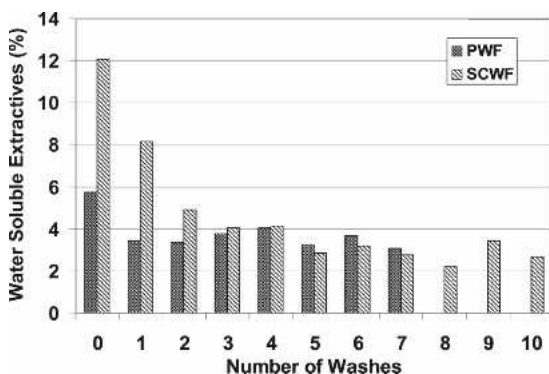


FIGURE 1. Water-soluble extractives remaining in pine wood-flour (PWF) and salt cedar wood-flour (SCWF) vs number of washes.

TABLE 3. Color parameters for wood flour subjected to two treatments, washing and coloring.

Wood-flour type	L*	a*	b*
PWF	78.7	4.5	22.0
PWF–Washed	71.7	4.8	19.9
SCWF	64.9	6.8	15.4
SCWF–Washed	59.0	4.5	14.4
PWF	80.4	4.8	23.6
PWF–S	43.7	4.7	5.3
PWF–D	37.8	2.3	0.9

PWF = pine wood-flour, SCWF = salt cedar wood-flour
S = stained, D = dyed

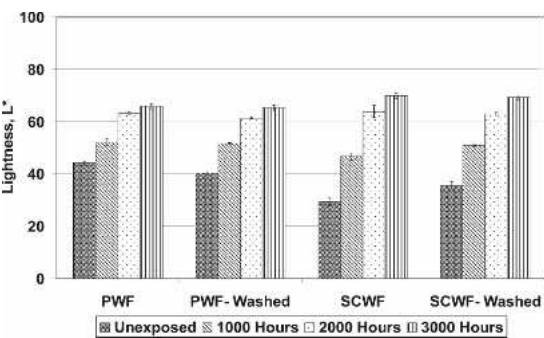


FIGURE 2. Lightness, L*, of composites containing 25% unwashed and washed pine wood-flour (PWF) and salt cedar wood-flour (SCWF) exposed to accelerated weathering.

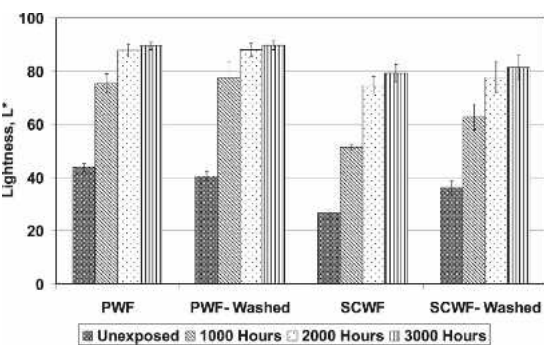


FIGURE 3. Lightness, L*, of composites containing 50% unwashed and washed pine wood-flour (PWF) and salt cedar wood-flour (SCWF) exposed to accelerated weathering.

Figures 2 and 3 show L* for composites containing 25 and 50% wood flour, respectively, before and during weathering. The SCWF composites were initially darker than the PWF. During accelerated weathering, the 25% wood-flour composites continued to lighten through 3000 h, while the 50% wood-flour composites lightened

through 2000 h before reaching a plateau. Composites containing more wood flour (50 vs 25%) lightened more. Comparing composites containing unwashed vs washed wood flour after weathering, the value of L* appeared similar.

The effect of fiber washing on L* is summarized in Table 4. After 3000 h of weathering, the L* of composites containing unwashed wood flour vs washed wood flour were not statistically different. This was true for both wood-flour content (25 and 50%) and both wood-flour type (PWF and SCWF). Therefore, despite conventional wisdom, washing water-soluble extractives using this procedure was found to be ineffective at improving the color stability of WPCs.

Colored wood flour

PWF was colored in a batch process using an oil-based stain or a water-based dye to manufacture stained wood flour (PWF-S) or dyed wood flour (PWF-D). After coloring, PWF-S was a dry, free-flowing, dark brown wood flour and PWF-D was a dry, free-flowing, dark brownish black wood flour. The color attributes are reported in Table 3. Both treatments resulted in a decrease in L* compared with untreated PWF (Table 3). It was clear that the PWF-D was darker than the PWF-S. Compared with untreated PWF, the color of PWF-S shifted away from yellow after treatment, while the color of PWF-D shifted away from both yellow and red.

Composites were manufactured by filling HDPE with PWF, PWF-S, or PWF-D. Additionally, composites were evaluated with and without a

TABLE 4. The effect of wood-flour washing on L* for wood-plastic composites after 3000 h of accelerated weathering.

	Unexposed		3000 h exposure	
	Unwashed	Washed	Unwashed	Washed
PWF, 25%	44.3 ^A	40.2 ^D	65.9 ^F	65.3 ^F
SCWF, 25%	29.4 ^B	35.6 ^E	69.9 ^G	69.2 ^G
PWF, 50%	43.9 ^A	40.5 ^D	89.5 ^H	89.7 ^H
SCWF, 50%	26.8 ^C	36.0 ^E	79.2 ^I	81.5 ^I

PWF = pine wood-flour, SCWF = salt cedar wood-flour
Different superscripts denote statistical differences at $\alpha = 0.05$

brown pigment incorporated into HDPE during compounding. Before exposure, WPCs manufactured with PWF-S and PWF-D were darker than WPCs manufactured with untreated PWF (Fig 4). The color was also visually more uniform, without individual wood particles visible. Weathering caused each composite to lighten. Composites containing PWF-S experienced a smaller increase in L^* , while composites containing PWF-D experienced a larger increase in L^* compared with composites containing untreated PWF. Adding pigment caused composites containing PWF and PWF-D to lighten less, while adding pigment to composites containing PWF-S did not have a large effect.

Initially, composites containing PWF-D were darker than those containing PWF-S and untreated PWF, respectively (Table 5). Adding pigment to the HDPE matrix decreased L^* for untreated PWF composites and slightly increased L^* for colored wood-flour composites. After weathering 3000 h, L^* was slightly higher for PWF-D composites, and much lower for PWF-S composites compared with untreated PWF composites. Adding pigment to composites resulted in smaller values of L^* after weathering compared with composites without pigment, indicating that pigment was beneficial to improving the color stability of WPCs. However, the benefit of adding pigment was larger for composites containing PWF or PWF-D; adding pigment to composites containing PWF-S resulted in only a small improvement. After

TABLE 5. The effect of wood-flour coloring on L^* for wood-plastic composites after 3000 h of accelerated weathering.

	Unexposed		3000 h Exposure	
	No Pigment	Pigment	No Pigment	Pigment
PWF	47.0 ^A	37.1 ^D	87.3 ^F	58.8 ^I
PWF-S	30.3 ^B	31.1 ^E	50.2 ^G	47.5 ^A
PWF-D	26.4 ^C	29.0 ^B	90.4 ^H	60.0 ^J

PWF = pine wood-flour, S = stained, D = dyed

Different superscripts denote statistical differences at $\alpha = 0.05$

weathering, composites containing PWF-S without pigmented HDPE performed better than those containing PWF with pigmented HDPE, the typical way of improving WPC color stability. This suggested that coloring wood particles using an oil-based stain could improve WPC color stability.

SUMMARY AND CONCLUSIONS

Two methods of treating wood flour, either by washing or by coloring before incorporation into WPCs, were evaluated as methods to improve the color stability of WPCs during weathering. The composite lightness, L^* , was monitored to quantify this relationship.

Although the water-soluble extractive content was initially higher for SCWF than PWF, washing the wood flour a number of times as described in this report resulted in similar final water-soluble extractive content. Washing slightly darkened each wood-flour species. Unwashed and washed PWF and SCWF were incorporated into WPCs at 25 and 50%. Initially, SCWF composites were darker than PWF composites. Weathering resulted in increases in L^* , and composites containing 25% wood flour lightened less than those containing 50% wood flour. Washing the wood flour, regardless of wood-flour type (SCWF or PWF) and content (25 or 50%), did not significantly change the color stability of WPCs.

Coloring PWF by either staining or dying resulted in colored wood flour that was darker than untreated PWF. Composites incorporating colored wood flour were also darker than compos-

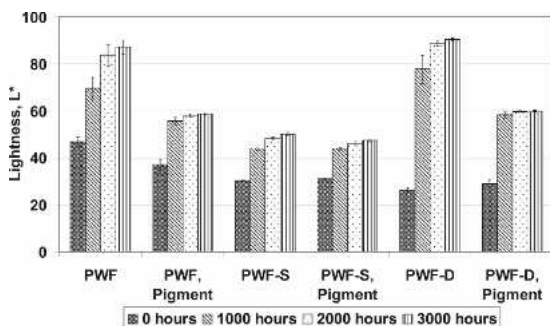


FIGURE 4. Lightness, L^* , of composites containing 50% pine wood-flour (PWF) used either as received, stained (-S) or dyed (-D) exposed to accelerated weathering.

ites incorporating untreated PWF. During weathering the composites containing PWF-S lightened less than those containing PWF, and composites containing PWF-D lightened more than those containing PWF. Adding pigment to the HDPE component of the composites containing PWF or PWF-D resulted in more color stability, while adding pigment to the HDPE component of the composites containing PWF-S only slightly improved color stability. Coloring wood flour using an oil-based stain was shown to be a promising method to improve the color stability of WPCs during weathering.

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