DEUTERIUM AS A NOVEL TRACER FOR DETERMINING MOISTURE SOURCES IN BUILDING SYSTEMS

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ABSTRACT

Many current problems in moisture-related deterioration of housing are found in the building envelope. Components such as interior surfaces, siding, millwork, sheathing, and framing are damaged due to excessive moisture accumulation, which leads to potential biodeterioration (mold and decay). The monetary and health implications of such widespread failures are severely impacting the housing industry. In certain individual cases of wood wetting, the source of liquid water is readily determined: typically, a failure of external sealing allows direct ingress of precipitation into the wall system. There is, however, still a debate on

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the role of subsequent moisture movement or condensation (from water vapor movement to a cool surface) in wall and roofing system failures. Here we show that deuterium-labeled water introduced into wood through either liquid or vapor phase can be recovered via distillation or mechanical expression. The use of deuterium as a moisture tracer can assist in determining the origin of moisture (particularly through the vapor phase transition), provide insight into current recommended practices for moisture control in buildings, and assess the application of current moisture modeling on existing moisture failures.

Keywords: Deuterium, stable isotope tracer, moisture, building systems

The trend toward building more energy efficient houses with vapor barriers, increased amounts of insulation, and new low-permeability siding systems has exacerbated the problem of excessive moisture in wall and ceiling systems. While these features are installed to prevent moisture from entering into the structural cavities (e.g., wood-framed walls), they also reduce the drying potential if they become wet by other means, such as moist air transport or water intrusion, which leads to potential biodeterioration (mold and decay) (Miller 1992). The monetary and health implications of such widespread failures are severely impacting the housing industry (Cheple 1999; Smulski 1999). This is especially true for cold climate buildings that traditionally have had a high probability for wintertime condensation on the warm side of exterior sheathing (Angell and Olson 1988; Burch and Thomas 1991; Hutcheon and Handegord 1989; Latta 1985; TenWolde 1994). Problems have also been reported in very hot and humid climates where air conditioners are used (Sherwood 1985). In certain individual cases of wood wetting, the source of liquid water is readily determined: typically, a failure of external sealing allows direct ingress of precipitation into the wall system. There is, however, still a debate on the role of subsequent moisture movement or condensation (from water vapor movement to a cool surface) in wall and roofing system failures (Lawton 1998; Smulski 1994). By the time a moisture problem is noticed, it is often difficult to ascertain the original source of moisture (external precipitation vs. internal condensation).

To address this problem, experiments were conducted using deuterium (D_2O)-enriched water as a moisture tracer in wood products. Deuterium (^2H), a stable (non-radioactive) isotope, has been used in cellulose studies to determine the accessibility and crystallinity of cellulose, and the water adsorption (Frilette et al. 1948; Knight et al. 1969; Mann and Marrinan 1956a–c; Taniguchi et al. 1978). Deuterium has an advantage over other tracers, such as lithium chloride (Back et al. 1978; Harris and Gale 1967a,b), by being able to transfer into wood through both the liquid and vapor phases. Condensation (liquid water) and vapor transport are two major avenues of moisture accumulation within wood-frame housing. Water can be present within the wood structure in two forms: adsorbed (or bound) water that is attached to hydroxyl groups on cellulose fibers at moisture contents (MC) below the fiber saturation point (fsp, ~25–30%); and capillary (or free) water present within the wood matrix at MC above the fsp. Moisture content is generally defined as the mass of water per mass of oven-dried wood, expressed as a percentage. The goal of this study was to determine recovery of deuterium-containing water from wood equilibrated with isotopically-labeled liquid or vapor, by removing moisture through toluene distillation (low or high MC) (Technical Association of the Pulp and Paper Industry 1996) or by mechanically dewatering the substrate with a hydraulic press (high MC) (Haygreen 1982). Minute quantities of water (4 μL) could then be analyzed for deuterium content (noted as δ^2H values, in per mil (‰) deviation relative to the Vienna Standard Mean Ocean Water (V-SMOW)), and compared to the deuterium content of the moisture source.

METHODOLOGY

Pinus ponderosa Laws. wood cubes (~1.01 cm per side) were used throughout the experiment. To eliminate sample variability due to density, the wood blocks were taken from the same wood sample board (sp. gr: χ = 0.433, σ =

0.005). D_2O-labeled water was added to the wood in liquid and vapor form (Fig. 1). Before liquid addition, all wood samples were oven-dried at 105±2°C until weight remained constant. During the vapor experiment, both oven- and air-dried specimens were used; the MC for the air-dried samples was ~4.09%.

Deuterium treatment and analysis

Deuterium (D_2O) (Aldrich Chem. Co. 19,234−1, sp.gr. = 1.11, 99.9%) was used as the tracer in all experiments. A concentration of 183 mg L^{-1} was necessary to create a tracer solution with a δ^2H value of approximately +1100 ‰ in order to remain within the range of the analytical equipment used for isotope analysis. The deuterium content of water was determined by chromium reduction. Four µL of sample was injected into a chromium-filled quartz reactor held at 960°C and evacuated to ~10^{-3} Torr. $H_{2\,(gas)}$ was produced and collected within 60 s. Subsequently, hydrogen isotope ratios ($^2H/^1H$) were determined by dual inlet isotope ratio mass spectrometry (Donnelly et al. 2001; Nelson and Dettman 2001). Internal laboratory standards (CW: Calgary water, OS: "old SMOW," SWAT) were calibrated periodically using international standards (V-SMOW, V-SLAP, V-GISP), and analyzed repeatedly within each sample set (1 standard per 5 samples) to guarantee quality control. Isotopically enriched water samples were calibrated using additional standards with positive δ^2H values provided by the International Atomic Energy Agency (IAEA); e.g., IAEA 302A and 302B. Corrected δ^2H values were reported in the per mil (‰) notation relative to V-SMOW. Accuracy and precision for δ^2H values of natural waters were generally better than ±2.0 ‰, and those of ^2H-enriched samples were better than ±6 ‰ (one standard deviation based on n=10 lab standards).

Condensation

A styrofoam container measuring 64 cm × 42 cm × 31 cm with 4-cm-thick walls was modified to collect condensation on a glass plane set in insulation between the heated water source and a cold source (Fig. 2). A hotplate/stirrer was placed on the "warm side" of the container. Rigid extruded polystyrene insulation foam (Foamular 150, 5 cm thick, R-value = 10) was modified to hold a pane of window glass (17.2 cm × 13.4 cm × 0.02 cm) at an angle of 15 degrees. The slope of approximately 0.29 across the length of the glass was necessary to drain the liquid condensate on the warm side of the glass. A 2-cm diameter polyvinyl chloride (PVC) pipe with a 5-mm-wide slot to receive the bottom edge of the glass was inserted through the styrofoam container and the insulation foam. Waterproof caulking was applied to the "cold side" of the glass at the junction of the PVC pipe to ensure that moisture entered the drainage pipe only

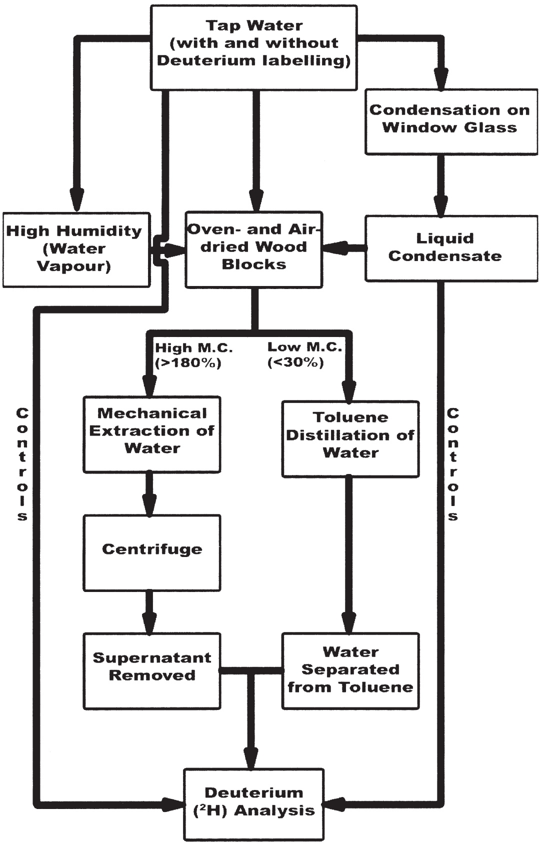

FIG. 1. Flow diagram for deuterium-labeled and non-labeled water treatments to wood and methods of recovery for analysis.

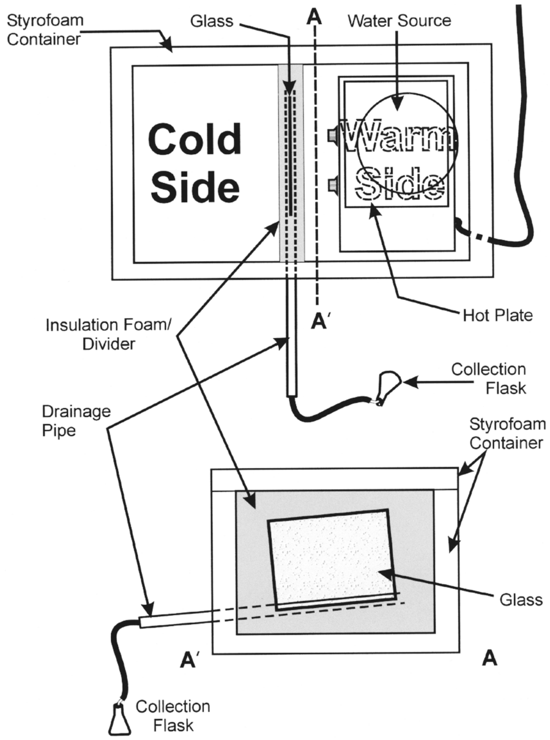

FIG. 2. Basic apparatus for generation and collection of deuterium-labeled condensate.

from the warm side. A hose connected the end of the PVC pipe to a collection flask.

To generate condensation, approximately 500 ml of water was placed in a 2500 ml Pyrex crystallizing dish and heated to 50°C on the hotplate while being stirred with a magnetic stir bar. A cold source was inserted on the cold side, and a lid was placed on the container. The vapor from the warm side of the unit condensed on the windowpane, and the liquid condensate ran through the drainage pipe and into the collection flask. The experiment was allowed to run between 18 and 24 h to collect 150 to 200 ml of liquid condensate.

Vapor

To generate a high humidity environment (R.H. $> 95\%$), the wood blocks were placed on a perforated tray in a sealed desiccator over the deuterium-labeled water. The entire unit was then placed in an oven at 50±2°C for 2 weeks to ensure vapor saturation. During the experiment, no liquid condensate was allowed access to the wood blocks. To test the effect of temperature on the accumulation of the deuterium tracer, the experiment was repeated using 12 samples at 30±2°C. Four of these samples were placed in a separate container with its own moisture source and measured at seven-day intervals until achieving a MC approaching fsp. This phase of the experiment ran for three weeks.

Liquid

Liquid water was added to the wood as D_2O-labeled water and D_2O-labeled condensation. To achieve a MC approaching saturation ($>180\%$), specimens were immersed in the deuterium-labeled water, a vacuum was applied to remove air, and then the vacuum was released to allow penetration of the liquid. For a MC near fsp (~30%), liquid water and condensate were added to the end grain of the wood samples as droplets to simulate condensation formation and uptake. After droplet addition, each wood specimen was placed in a paraffin-film covered 50-ml beaker (65 cm^3) and allowed to equilibrate for 72 h. Both experiments were carried out at an ambient temperature of 30±2°C.

Mechanical expression

A portable stainless steel piston assembly consisting of an open-ended cylinder, a separate stainless steel base with a rubber gasket to receive the bottom of the cylinder, and a removable piston head with a ram face diameter of 6.4 cm was used to compress the wood. Fluorinated ethylene propylene (FEP, Cole Parmer) tubing was used to convey the liquid water from a small opening at the bottom of the outer cylinder to the collection flask, and to connect the collection flask to the vacuum pump. The entire assemblage was then moved to the hydraulic press (Carver Laboratory Press, Model B) for compression, and the collection tube was inserted into the cylinder. A vacuum of 51–68 kPa (380–510 Torr) was applied to the cylinder, and

67 kN was applied to the wood block for 2 min. After 2 min, the force was released momentarily, and then 67 kN was reapplied to the compressed wood block for an additional minute. The 67 kN translates to a pressure of 21.1 MPa at the ram face of the piston.

The water was centrifuged in a Clay Adams Safety Head centrifuge at high speed for 30 min to separate the water from the particulate matter in solution. The supernatant was transferred to a small vial, sealed with paraffin film, and submitted for isotope analysis.

Toluene distillation

Toluene distillation (TAPPI method T 208 om-94) was used to remove water from wood samples with low moisture contents (Technical Association of the Pulp and Paper Industry 1996). The wood blocks were split into quarters to fit through the mouth of the round-bottomed boiling flask filled with toluene ($C_6H_5CH_3$, Fisher Scientific T290−4, HPLC Grade). The resultant water vapor from the samples boiled in toluene was condensed in a Liebig-type condenser and collected in a distilling receiver until the water level did not change for 15 min. A drying bulb filled with desiccant was placed at the top of the condenser to preclude outside moisture from contaminating the sample during cooling. After cooling, the water and a small amount of toluene were removed from the distilling receiver with a glass pipette and placed in a sample vial. Water without the toluene was then transferred to a second sample vial with a glass pipette. The sample vial was capped, sealed with paraffin film, and sent for isotope analysis.

RESULTS AND DISCUSSION

The experiments were run three separate times (Run 1−3). Runs 1 and 2 used separate D_2O-labeled source water, and encompassed the liquid (both saturated and droplet) and vapor experiments. Run 3 used the same deuterium-labeled source liquid as in Run 2, but only for the vapor phase. The third run was performed to test the effect of temperature (30°C as opposed to 50°C) on vapor saturation. Tap water collected for comparison had δ^2H values of ~ –48 ‰, while the source water (including condensation) used in all phases of the experiment had d^2H values of ~ +1100 ‰ (Table 1). The results are expressed as a ratio of the deuterium content in the water recovered from the wood ($\delta^2H_{Recovered}$) to the deuterium content present in the source liquid (δ^2H_{Source}). Control D_2O-labeled source water was analyzed after toluene distillation to determine if deuterium was lost during the process. Similarly, control samples were collected and tested after vacuum and vapor saturation of the wood blocks. There was some variability in δ^2H values of the liquid when compared to the condensate in Run 1, but the difference was very small. Likewise, there was only a minor loss of D_2O during toluene-distillation. The largest variation in δ^2H values occurred during the vacuum saturation of the wood blocks. The majority of groups showed minor losses of deuterium in source water after vacuum impregnation of the wood blocks. An exception to this was the minor increase in deuterium content of one group during Run 1. This deuterium deletion and enrichment may be caused by water vapor exchange and/or evaporation into the atmosphere during the experiment.

Table 2 shows deuterium contents of water recovered from wood blocks. There was no significant difference ($\alpha = 0.05$) between the D_2O-labeled liquid and condensate removed from the wood, suggesting that the condensation process does not affect deuterium uptake within the samples. Similarly, there was little difference between the oven-dried and air-dried specimens, indicating that deuterium-containing H_2O replaces any moisture present within the wood. The results were grouped together by addition method (saturation, droplet, and vapor) for the three runs, irrespective of source liquid or moisture content of the wood samples (Fig. 3). There was almost complete recovery of the deuterium-labeled water from the saturated liquid and vapor groups, while the samples exposed to the liquid droplets showed a 35% drop in δ^2H values. Tem-

TABLE 1. *Results from hydrogen isotope ratio analysis of deuterium-labeled water used during the experiment.*

						δ^2H (‰)	$\delta^2H_{Recovered}$ (‰)/ δ^2H_{Source} (‰)	
Run	Temp (C)	Source	Form[a]	Extraction[b]	n	χ	χ	σ
1	30	D_2O	Liquid	None	4	1086	N/A	0.00
1	30	D_2O	Condensate	None	4	1113	1.02	0.00
1	30	D_2O	Condensate	Aft vacuum	4	1094	0.98	0.00
1	30	D_2O	Liquid	Aft vacuum	4	1110	1.02	0.00
1	30	D_2O	Liquid	Distill	4	1068	0.98	0.03
1	50	D_2O	Liquid	Aft vapor	4	1071	0.99	0.00
2	30	D_2O	Liquid	None	4	1077	N/A	0.00
2	30	D_2O	Condensate	None	4	1076	1.00	0.001
2	30	D_2O	Condensate	Aft vacuum	4	1047	0.97	0.01
2	30	D_2O	Liquid	Aft vacuum	4	1033	0.96	0.01
2	30	D_2O	Liquid	Distill	4	1070	0.99	0.00
2	50	D_2O	Liquid	Aft vapor	4	1059	0.98	0.00
3	30	D_2O	Liquid	None	4	1080	N/A	
3	30	D_2O	Liquid	Aft vapor	4	1067	0.99	0.00

[a] Condensate refers to the liquid condensate generated during the experiment (see Fig. 2).

[b] Aft Vacuum refers to the liquid left in the beaker after vacuum saturation of the specimens; Aft Vapor refers to the liquid remaining after vapor saturation of the specimens; Distill refers to toluene distillation of the source liquid

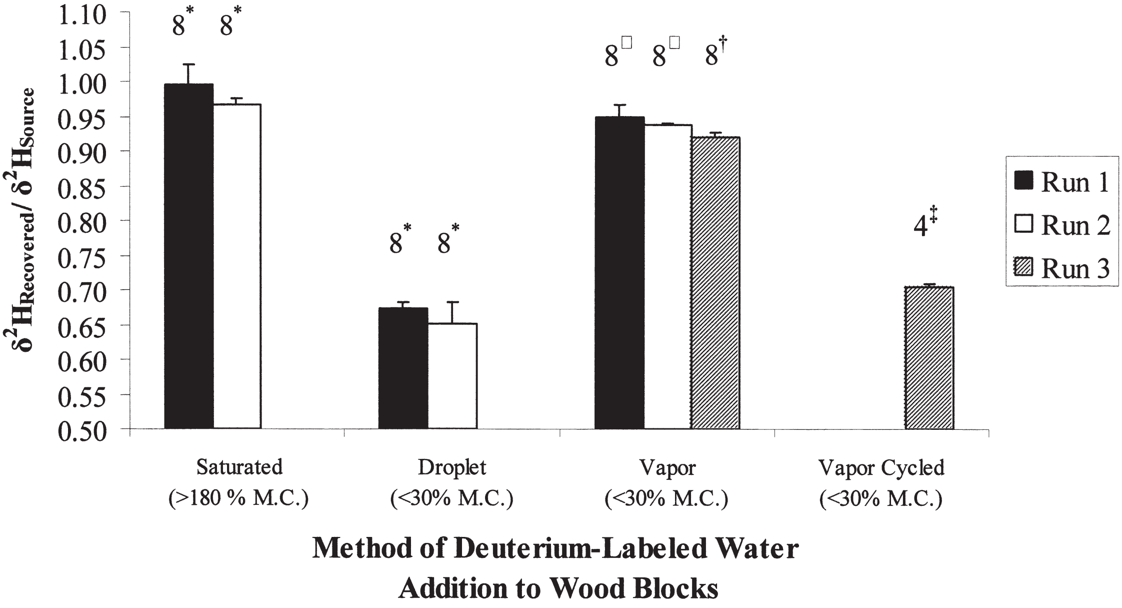

FIG. 3. Recovery of deuterium-labeled water from *P. ponderosa* wood blocks. Error bars represent one standard deviation from the group mean. Numbers above error bars give sample sizes for the group. Symbols: *—sample group includes both deuterium-labeled water and condensate; □—sample group includes deuterium-labeled water from air-dried and oven-dried specimens, vapor was generated at 50±1 C; †—sample group includes deuterium-labeled water from oven-dried specimens, vapor was generated at 30±2 C; ‡—sample group includes deuterium-labeled water from oven-dried specimens, vapor was generated at 30±2 C, samples were repeatedly weighed and exposed to non-labeled moisture in the air.

TABLE 2. *Results from hydrogen isotope ratio measurements of deuterium-labeled water extracted from* P. ponderosa *wood blocks.*

								δ^2H (‰)	$\delta^2H_{Recovered}$ (‰)/ δ^2H_{Source} (‰)	
Run	Wood drying	Temp (C)	Source	Form	Addition	Extraction	n	χ	χ	σ
1	Oven-dried	30	D_2O	Condensate	Vacuum	Mech	4	1105	0.99	0.00
1	Oven-dried	30	D_2O	Liquid	Vacuum	Mech	4	1083	1.00	0.05
1	Oven-dried	30	D_2O	Condensate	Droplet	Distill	4	753	0.68	0.01
1	Oven-dried	30	D_2O	Liquid	Droplet	Distill	4	729	0.67	0.01
1	Air-dried	50	D_2O	Vapor	Vapor	Distill	4	1038	0.96	0.00
1	Oven-dried	50	D_2O	Vapor	Vapor	Distill	4	1025	0.94	0.02
2	Oven-dried	30	D_2O	Condensate	Vacuum	Mech	4	1040	0.97	0.01
2	Oven-dried	30	D_2O	Liquid	Vacuum	Mech	4	1040	0.97	0.01
2	Oven-dried	30	D_2O	Condensate	Droplet	Distill	4	705	0.66	0.03
2	Oven-dried	30	D_2O	Liquid	Droplet	Distill	4	695	0.65	0.03
2	Air-dried	50	D_2O	Vapor	Vapor	Distill	4	1010	0.94	0.00
2	Oven-dried	50	D_2O	Vapor	Vapor	Distill	4	1008	0.94	0.00
3	Oven-dried	30	D_2O	Vapor	Vapor	Distill	4	995	0.92	0.01
3	Oven-dried	30	D_2O	Vapor	Vapor	Distill	4	758	0.70	0.00
3[b]	Oven-dried	30	D_2O	Vapor	Vapor	Distill	4	758	0.70	0.00

[a] Source for Liquid and Vapor is the mean δ^2H (‰) value for liquid without extraction; Source for Condensate is the mean δ^2H (‰) value for condensate without extraction (see Table 1).

[b] Represents samples that were weighed repeatedly to verify vapor saturation.

perature had no effect on the retrieval of deuterium from the samples during the vapor experiments.

Currently we do not have an explanation for the 35% drop in deuterium content of water retrieved from these wood samples, but we are running experiments to ascertain the source of the loss. There may be fundamental differences in the way the vapor and liquid phases act within the wood structure. Experiments conducted on regenerated cellulose (cellulose II) suggest that liquid deuterium may become bound to the surface of the crystalline cellulose structure, while ^2H-containing vapor predominantly attaches to the more accessible amorphous cellulose (Mann and Marrinan 1956a–c). While this may be part of the explanation, the exposure of the wood specimens to water molecules in the air is more likely. A 30% drop in δ^2H values also occurred in vapor-exposed wood samples (30°C) that were repeatedly removed and weighed to ascertain MC during Run 3. The deuterium-labeled source water was not replenished during the three-week time span of the experiment. Non-labeled water molecules from the air may have exchanged with deuterium-labeled molecules in the wood during weighing. This same process would explain the decrease in δ^2H values of the liquid droplet groups: while the volume of air within the beakers was small, displacement of the labeled-water molecules probably occurred. Neither the saturated wood specimens nor blocks equilibrated to lower equilibrium moisture content (EMC) in a D_2O-labeled vapor atmosphere showed this characteristic drop.

CONCLUSIONS

The data clearly demonstrate that sufficient quantities of deuterium-labeled water can be recovered from the wood samples to show the labeled origin of moisture. For most experiments, the deuterium content of the recovered water was almost identical to that of the labeled source water (i.e., ratio ~1). The only data groups for which lower than expected deuterium contents were observed in the recovered water (i.e., ratio <1) were those that had water added via droplets and vapor saturated samples repeatedly exposed to the atmosphere for weighing. It is hypothesized that after initial uptake of deuterium-labeled water, wood specimens exposed to atmospheric moisture may show a decrease in δ^2H values of the recovered water due to water molecule exchange. While the drop in deuterium content is significant (30–35%), experimental data indicate that δ^2H values ($> +700$ ‰) remain far in excess to those found in tap water (~ –48 ‰) or unlabeled atmospheric moisture. The results provide researchers with a new tool to trace moisture in building systems from the liquid phase, through vapor phase transition, to accumulation via condensation or vapor in building components. Such application may assist in the development of improved moisture modeling in current building systems (Burch 1992; Desjarlais and Byars 1998; TenWolde 2001) or design and material changes for improved moisture control (American Society for Testing and Materials 2001; Lstiburek and Carmody 1993).

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