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# Comparison of the saturated salt and dynamic vapor sorption methods in obtaining the sorption properties of *Pinus pinea* L.

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Abstract Several methods are available for obtaining the sorption isotherms of wood. Among these, the saturated salt and dynamic vapor sorption methods are the most frequently used ones. For the first time, the hygroscopic response of wood obtained using these two methods is compared. This is done by determining the 35 and 50 °C adsorption isotherms of juvenile and mature wood of Pinus pinea L. The hygroscopic behavior of the two types of wood is different, as the mature wood has a higher moisture content than the juvenile wood in the isotherms studied. Comparison of the static saturated salt method and dynamic vapor sorption shows few significant differences between the equilibrium moisture content obtained by each method during the adsorption process, both in a point by point comparison and in the comparison of quadratic polynomial forms of the Guggenheim Anderson-de Boer model. Moreover, in both methods the point of relative humidity from which multilayer sorption predominates over monolayer sorption is similar.

## 1 Introduction

Numerous studies have been conducted on the hygroscopic behavior of wood. The sorption isotherms of various species have been determined (Popper et al. 2009; Hill et al. 2010; Hoffmeyer et al. 2011; Zaihan et al. 2011; Fernandez et al. 2014), also differentiating between sapwood and heartwood (Ball et al. 2001; Telkki et al. 2013; Song et al. 2014) and between juvenile and mature wood (Lenth and Kamke 2001; Militz et al. 2003; Neimsuwan et al. 2008; Hill et al. 2011; Esteban et al. 2015; Simon et al. 2015). Other authors obtained the isotherms of wood that was modified thermally (Engelund et al. 2010; Jalaludin et al. 2010; Hill et al. 2012; Murata et al. 2013; Pearson et al. 2013) or chemically (Cao and Kamdem 2004; Dieste et al. 2010; Engelund et al. 2010; Xie et al. 2011; Popescu et al. 2014), and also of old wood (Esteban et al. 2006, 2008a, 2010; Bratasz et al. 2012; Popescu and Hill 2013). This variety of studies confirms the importance of knowing the hygroscopic behavior of wood and its repercussion on the physical and mechanical properties of this material (Skaar 1988; Siau 1995; Almeida and Hernandez 2006; Hill 2006; Kretschmann 2010; Rautkari et al. 2014).

The literature includes various methods for obtaining the equilibrium moisture content (*EMC*) in wood. Among these, the static gravimetric method of saturated salts has been widely used (Cao and Kamdem 2004; Almeida and Hernandez 2006; Esteban et al. 2008b; Majka and Olek 2008; Thygesen et al. 2010; Hoffmeyer et al. 2011; Murata et al. 2013; Olek et al. 2013; Fernandez et al. 2014). This method, described in COST Action E8, consists of keeping the samples in a container with saline solutions that generate known relative humidities (*RH*) until the wood reaches a state of equilibrium with the ambient *RH*. Samples are weighed at regular intervals until they reach a constant value corresponding to the equilibrium state.

However, new studies conducted increasingly use dynamic vapor sorption (DVS) (Engelund et al. 2010; Hill et al. 2010, 2012; Jalaludin et al. 2010; Xie et al.

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2011; Zaihan et al. 2011; Popescu and Hill 2013; Popescu et al. 2014). This new technology generates the air relative humidity values by mixing dry nitrogen or air with saturated water vapor in automatically controlled proportions. The material is placed inside a small chamber where, as a result of a continuous flow of nitrogen and saturated water vapor over the sample, the equilibrium conditions of the material are reached in less time (Bingol et al. 2012). Changes in mass are constantly and automatically recorded.

The advantages of DVS over the saturated salt method are: (1) faster data acquisition (Engelund et al. 2011)-DVS technique requires a few days to determine a sorption isotherm whereas the salt method using desiccators takes months; (2) greater accuracy, as an automatic balance is used instead of a manual balance (Jalaludin et al. 2010); (3) continuous values for changes in mass are available as opposed to the discrete data of the static method (Zaihan et al. 2009), making it possible to monitor the process in real time and study changes over time; (4) the kinetic parameters of the sorption process that is related to the coefficients of diffusion, can be determined (Popescu et al. 2014) and (5) the possibility of obtaining all the values in the range of 0-96% RH (Engelund et al. 2011). The major disadvantage of DVS compared to the salt method is the high cost of the equipment (Arlabosse et al. 2003).

Very few authors have compared the adsorption behavior obtained using the saturated salt method and DVS. Levoguer and Williams (1999) and Surface Measurement Systems Ltd. (2012) determined the EMC in microcrystalline cellulose reference material (MCC RM 302) through DVS and compared the values with those obtained using the COST 90 procedure. The results showed similarities between the two methods for RH values above 33%. Below this value, the EMC values were higher when obtained by DVS than by the salt method. Arlabosse et al. (2003) determined this comparison in microcrystalline cellulose (MCC), pharmaceutical granules, PolyEtherBlocAmide (PEBA) membrane and sewage sludges. The EMC obtained showed small differences between the methods, which were more pronounced when the apparent diffusion coefficient in the material was higher than  $10^{-9}$  m<sup>2</sup>/s. Paes et al. (2010) studied the two methods in samples of ball milled cellulose and concluded that they give similar results. Bingol et al. (2012) obtained the sorption behavior in two varieties of rice using both methods and established that they have similar behavior. None of these studies used wood as the material.

The objective of this study was to compare the saturated salt and dynamic vapor sorption methods for determining the hygroscopic behavior of wood, using the adsorption isotherms of juvenile and mature wood of *Pinus pinea* L. to obtain the degree of similarity of the results.

### 2 Materials and methods

The samples of *Pinus pinea* L. were collected in the municipalities of Coca and Navas de Oro, in the province of Segovia (Spain). All the trees were adults more than 70 years old. A radial slice was taken from the basal disc, obtained 50 cm from the ground. Samples were taken from the juvenile and mature wood zones, defining the juvenile zone as the fraction between rings 10–20 and the mature wood zone as the area with rings older than 50 years. All the samples chosen were from the heartwood, both early-wood and latewood.

## 2.1 Saturated salt method

The isotherms were obtained at 35 and  $50 \,^{\circ}$ C following the methodology described by COST Action E8 (Themelin et al. 1997). The dimensions of the test pieces for both juvenile and mature wood were 15 mm long, 10 mm wide and 1 mm thick, with no specific anatomical orientation. The samples used in the tests had not been through a drying cycle.

The isotherms were plotted using the 10 equilibrium points corresponding to 10 different salts (Table 1). The relative humidity values generated by each salt for each temperature (35 and 50 °C) were taken from Stokes and Robinson (1949), Robinson and Stokes (1959) and Wexler and Wildhack (1965). Three replicates were performed per study sample.

The test pieces were then placed in a desiccator with phosphorous pentoxide for 40 days to anhydrous state. Then the adsorption isotherms were plotted. The samples were considered to have reached equilibrium in each salt when the difference in mass was no more than 0.1% when they were weighed in a 24 h interval. This process took 1 month.

## 2.2 Dynamic vapor sorption

The sorption isotherms were determined using the DVS-Advantage instrument. The wood was ground in a Retsch SM 2000 mill and sifted to improve air circulation. The size of the particles used in the experiment was 0.5–1.0 mm. The isotherms were determined at 35 and 50 °C. The *RH* used was the same as in the static saturated salt method. Because of the high reproducibility of the instrument (Jalaludin 2012), only one sample was used per measurement. The initial mass of the sample was 20 mg. The sample was placed in the DVS instrument and dried to obtain the dry mass, then hydrated to reach the corresponding *RH*. The sample was considered to have reached equilibrium when the change in mass (dm/dt) was less than 0.001% min<sup>-1</sup> for 10 min. The change in mass was recorded every minute. **Table 1** Equilibrium moisturecontents for 35 and 50 °C

isotherms

Salt	35 °C Is	sotherm				50°C Is	sotherm			
	RH	Juvenile	e	Mature		RH	Juvenil	e	Mature	
		Salt	DVS	Salt	DVS		Salt	DVS	Salt	DVS
LiCl	11.17	2.29 0.18 <sup>a</sup>	2.16	2.54 0.12 <sup>a</sup>	2.30	11.05	2.07 0.16 <sup>a</sup>	2.09	2.24 0.12 <sup>a</sup>	2.30
CH <sub>3</sub> COOK	21.37	3.05 0.23 <sup>a</sup>	3.07	$3.22 \\ 0.16^{a}$	3.47	20.06	$2.55 \\ 0.20^{a}$	3.02	3.01 0.18 <sup>a</sup>	3.32
MgCl <sub>2</sub>	32.00	4.11 0.42 <sup>a</sup>	4.20	4.32 0.18 <sup>a</sup>	4.72	30.54	3.47 0.16 <sup>a</sup>	4.03	3.81 0.28 <sup>a</sup>	4.47
K <sub>2</sub> CO <sub>3</sub>	42.55	4.82 0.23 <sup>a</sup>	5.28	4.93 0.23 <sup>a</sup>	5.93	40.91	4.68 0.21 <sup>a</sup>	5.01	$4.79 \\ 0.18^{a}$	5.53
Mg(NO <sub>3</sub> ) <sub>2</sub>	49.72	6.11 0.09 <sup>a</sup>	5.98	6.83 0.10 <sup>a</sup>	6.69	45.44	$5.24 \\ 0.16^{a}$	5.40	5.43 0.42 <sup>a</sup>	5.94
SrCl <sub>2</sub>	66.08	8.27 0.35 <sup>a</sup>	8.00	9.41 0.42 <sup>a</sup>	8.88	57.46	$7.14 \\ 0.56^{a}$	6.62	$7.65 \\ 0.12^{a}$	7.40
NaCl	75.11	10.10 0.28 <sup>a</sup>	9.33	10.92 0.25 <sup>a</sup>	10.39	74.84	$8.89 \\ 0.27^{a}$	8.82	9.94 0.34ª	9.87
KCl	82.95	11.60 0.45 <sup>a</sup>	10.79	12.41 0.22ª	12.00	81.20	10.38 0.32 <sup>a</sup>	9.72	11.54 0.37 <sup>a</sup>	10.98
BaCl <sub>2</sub>	89.40	12.37 0.36 <sup>a</sup>	12.35	14.23 0.62 <sup>a</sup>	13.89	88.23	12.01 0.47 <sup>a</sup>	11.19	13.08 0.75 <sup>a</sup>	12.66
K <sub>2</sub> SO <sub>4</sub>	96.71	15.40 0.42 <sup>a</sup>	15.15	18.08 1.08 <sup>a</sup>	17.24	95.82	14.53 1.29 <sup>a</sup>	13.42	16.11 0.47 <sup>a</sup>	15.50

RH relative humidity

<sup>a</sup>Standard deviation

In both methods the equilibrium moisture content was determined using the following equation:

$$EMC(\%) = \frac{W_W - W_0}{W_0} \times 100$$
(1)

 $W_w$ : wet mass (g),  $W_0$ : anhydrous mass (g).

The isotherms were fitted using the Guggenheim Anderson-de Boer (GAB) model, which has been widely used to model the sorption isotherms of wood (Kelsey 1957; Themelin et al. 1997; Hartley 2000; Esteban et al. 2008a, 2010; Lesar et al. 2009; Todoruk and Hartley 2011; Bratasz et al. 2012; Lam et al. 2012; Olek et al. 2013). It also allows a type II sorption curve to be drawn in the whole *RH* range (Hartley 2000; Esteban et al. 2008a, 2010):

$$X = \frac{KC_g(RH/100)}{(1 - K(RH/100))(1 - K(RH/100) + C_gK(RH/100))}X_m$$
(2)

*X: EMC* (%),  $X_m$ : monolayer saturation moisture content (%),  $C_g$ : Guggenheim constant (dimensionless), *K*: constant (dimensionless), *RH*: relative humidity (%)

The isotherm fits were considered valid when the correlation coefficient (R) was greater than 0.990 and the root

mean square error (*RMSE*) was less than 4% (Viollaz and Rovedo 1999; Esteban et al. 2006, 2009).

#### 2.3 Comparison of methods

The methods were compared in two ways. Firstly, the existence of significant differences between the DVS measurement and the interval of variation of the measurement with the static saturated salt method was analyzed, using a twotailed test (Miller and Miller 2002) with a confidence level of 95%.

Secondly, the quadratic polynomial forms of the GAB equations of the methods were compared using the MAT-LAB program<sup>®</sup> Version 7.5.0 Curve Fitting Toolbox Version 1.2, which enables the confidence intervals of the fitted curves to be obtained.

The GAB equation is expressed in quadratic polynomial form as follows:

$$\frac{RH/100}{X} = \frac{1}{X_m}$$
$$\cdot \frac{(1 - K(RH/100))(1 - K(RH/100) + C_g K(RH/100))}{KC_g}$$



Fig. 1 35 and 50 °C sorption isotherms of juvenile and mature wood of *Pinus pinea* 

Table 2Results of the GAB fitfor the 35 and 50 °C isothermswith the saturated salt method

and DVS

$$y = A + Bx - Cx^2 \tag{3}$$

$$y = \frac{RH/100}{X}; x = RH/100; A = \frac{1}{X_m K C_g}; B = \frac{(C_g - 2)}{X_m C_g};$$
$$C = \frac{(C_g - 1)K}{X_m C_g}$$

This equation concurs with the polynomial form of the Hailwood–Horrobin model, which is also commonly used to model the isotherms of wood:

$$m = \frac{18}{W} \cdot \left[ \frac{k_1 k_2 (RH/100)}{1 + k_1 k_2 (RH/100)} + \frac{k_2 (RH/100)}{1 - k_2 (RH/100)} \right]$$
$$y = A + Bx - Cx^2$$
$$RH/100 \qquad \qquad W \qquad 1$$

$$y = \frac{KH/100}{X}; x = RH/100; A = \frac{W}{18} \cdot \frac{1}{k_2(k_1+1)};$$
$$B = \frac{W}{18} \cdot \frac{k_1 - 1}{(k_1+1)}; C = \frac{W}{18} \cdot \frac{k_1 k_2}{(k_1+1)}$$

Parameter	Juvenile		Mature	
	35 °C Isotherm	50 °C Isotherm	35 °C Isotherm	50°C Isotherm
Salt				
X <sub>m</sub> (%)	$4.47 \pm 0.25$	$4.40 \pm 0.31$	$4.58 \pm 0.33$	$4.41 \pm 0.26$
Κ	$0.76 \pm 0.39$	$0.75 \pm 0.44$	$0.79 \pm 0.53$	$0.78 \pm 0.41$
Cg	$7.91 \pm 0.06$	$6.57 \pm 0.09$	$8.21 \pm 0.08$	$7.72 \pm 0.07$
R	0.995	0.997	0.995	0.996
RMSE (%)	1.22	1.71	2.01	1.95
RH (%) <sup>a</sup>	33.00	32.80	31.70	31.90
EMC (%) <sup>a</sup>	4.32	3.99	4.47	4.22
EMC <sub>f</sub> (%)	0.15	0.42	0.11	0.18
DVS				
$X_{m}$ (%)	$4.63 \pm 0.10$	$4.63 \pm 0.05$	$5.23 \pm 0.06$	$5.03 \pm 0.06$
Κ	$0.73 \pm 0.15$	$0.70 \pm 0.08$	$0.73 \pm 0.09$	$0.72 \pm 0.08$
Cg	$7.75 \pm 0.02$	$8.21 \pm 0.01$	$7.38 \pm 0.02$	$8.15 \pm 0.01$
R	0.997	0.998	0.996	0.997
RMSE (%)	0.29	0.09	0.13	0.10
RH (%) <sup>a</sup>	34.10	35.70	34.10	34.80
EMC (%) <sup>a</sup>	4.44	4.52	4.95	4.90
EMC <sub>f</sub> (%)	0.19	0.11	0.28	0.13

 $X_m$  monolayer saturation moisture content, K constant,  $C_g$  Guggenheim constant, R correlation factor, RMSE root medium square error, RH relative humidity, EMC equilibrium moisture content, EMC<sub>f</sub> water taken up via monolayer sorption after the point of inflexion

<sup>a</sup>Point of inflexion of the isotherm



**Fig. 2** Point by point comparison of the saturated salt method and DVS. **a** Juvenile wood at 35 °C. **b** Mature wood at 35 °C. **c** Juvenile wood at 50 °C. **d** Mature wood at 50 °C

## 3 Results and discussion

Table 1 shows the *EMC* values of juvenile and mature wood for the static saturated salt method and the dynamic method (DVS). In all cases the isotherms describe a type II sigmoid (Fig. 1) characteristic of cellulosic materials

(Avramidis 1997; Zaihan et al. 2011; Engelund et al. 2013). *EMC* values are higher in salts than in DVS for high *RH* and lower for the remaining *RH*, except in the juvenile and mature wood at 11.17% *RH* and 35 °C (Table 1). The most likely explanation for this is that the ambient conditions for high *RH* are obtained with greater accuracy with DVS than with saturated salts.

Table 1 shows the relation between the sorption behavior of juvenile and mature wood. In all cases the *EMC* values are higher in the mature wood than in the juvenile wood and therefore there is a relation between growth ring location and sorption behavior. Other authors obtained similar results (Zobel et al. 1968; Militz et al. 2003; Neimsuwan et al. 2008; Esteban et al. 2015; Simon et al. 2015). In contrast, Lenth and Kamke (2001), Majka and Olek (2008) and Hill et al. (2015) obtained higher *EMC* values in juvenile wood than in mature wood. The difference in this behaviour is mainly due to the chemical composition of each type of wood (Esteban et al. 2015; Simon et al. 2015).

The isotherm fits with the GAB model are valid, as they have *R* values greater than 0.990 and *RMSE* values less than 4% for all cases (Table 2). The saturation moisture content of the monolayer ( $X_m$ ) is higher in the mature wood than in the juvenile wood at both temperatures. The *RH* values where multilayer sorption starts to predominate over monolayer sorption are at around 30% (Table 2), supporting observations in other species (Esteban et al. 2009, 2010, 2015; Fernandez et al. 2014). The similar value using the saturated salt method and DVS indicates the good correlation between the methods for obtaining the *EMC*.

Figure 2 shows the experimental *EMC* of DVS and the intervals of variation of the measurements with the saturated salt method.

A good correlation between salts and DVS was obtained, confirming the findings of other studies on different materials (Levoguer and Williams 1999; Arlabosse et al. 2003; Paes et al. 2010; Bingol et al. 2012; Surface Measurement Systems Ltd. 2012). There is only one *RH* value for which the methods differ. In the mature wood, at both 35 and 50 °C, this difference occurs for *RH* at around 40%, and in the juvenile wood it depends on the temperature, as it is 75% *RH* at 35 °C and 30% at 50 °C. Bingol et al. (2012) compared the *EMC* obtained with salts and with DVS in various forms of two varieties of rice. The differences between the two methods occurred in different *RH* ranges depending on the form of rice—in some cases for 0 and 98% *RH* and in others for *RH* around 40–80%. Therefore, it is not possible to establish a specific range of differences.

Table 3 shows the values of the quadratic polynomials of the GAB model using the saturated salt method and DVS. It was observed that the values of the two methods are similar

	RH (%)	JUVENILE					MATURE				
		SALT		DVS		X <sub>Salt</sub> ≠X <sub>DVS</sub>	SALT		DVS		$x_{Salt} \neq x_{DV}$
		RH/EMC (%)	$x (x_1 - x_2)$	RH/EMC (%)	$x (x_1 - x_2)$		RH/EMC (%)	x (x <sub>1</sub> - x <sub>2</sub> )	RH/EMC (%)	$x (x_1 - x_2)$	
ပ္ရ	11.17	4.88	5.42 (4.50-6.33)	5.17	5.43 (5.07–5.78)	1	4.40	5.03 (3.87-6.18)	4.86	4.95 (4.73–5.18)	I
	21.37	7.01	6.63 (6.03–7.23)	6.96	6.61 (6.37–6.84)	I	6.64	6.21 (5.45–6.97)	6.16	5.97 (5.83–6.12)	Ι
	32.00	7.79	7.57 (7.06–8.07)	7.62	7.53 (7.33–7.73)	I	7.41	7.11 (6.47–7.74)	6.78	6.77 (6.64–6.89)	I
	42.55	8.83	8.17 (7.62–8.71)	8.06	8.14 (7.93–8.35)	I	8.63	7.66 (6.97-8.34)	7.18	7.29 (7.15–7.42)	I
	49.72	8.14	8.38 (7.81-8.95)	8.31	8.38 (8.15-8.60)	I	7.28	7.84 (7.12–8.56)	7.43	7.49 (7.35–7.63)	I
	66.08	7.99	8.32 (7.78-8.85)	8.26	8.39 (8.18-8.60)	I	7.02	7.67 (6.99–8.34)	7.44	7.48 (7.35–7.61)	I
	75.11	7.44	7.94 (7.46–8.42)	8.05	8.08 (7.89–8.27)	I	6.88	7.23 (6.62–7.83)	7.23	7.20 (7.08–7.32)	I
	82.95	7.15	7.42 (6.93–7.90)	7.69	7.63 (7.44–7.82)	I	6.68	6.64 (6.03–7.26)	6.91	6.80 (6.68–6.92)	I
	89.40	7.23	6.85 (6.26–7.44)	7.24	7.13 (6.90–7.36)	I	6.28	6.02 (5.27–6.78)	6.44	6.36 (6.21-6.50)	I
	96.71	6.28	6.06 (5.23–6.88)	6.38	6.43 (6.11–6.75)	I	5.35	5.17 (4.13-6.21)	5.61	5.73 (5.53–5.94)	I
ç	11.05	5.34	6.16 (5.05–7.28)	5.29	5.40 (5.22–5.58)	I	4.93	5.44 (4.52-6.35)	4.80	4.90 (4.71–5.08)	I
	20.06	7.87	7.18 (6.43–7.93)	6.64	6.50 (6.38–6.62)	I	6.66	6.52 (5.90–7.14)	6.04	5.90 (5.77-6.02)	I
	30.54	8.80	8.07 (7.46-8.67)	7.58	7.51 (7.41–7.61)	I	8.02	7.47 (6.97–7.96)	6.83	6.81 (6.71-6.91)	*
	40.91	8.74	8.63 (7.98–9.29)	8.17	8.22 (8.11–8.32)	I	8.54	8.07 (7.53-8.61)	7.40	7.44 (7.33–7.55)	I
	45.44	8.67	8.78 (8.10–9.47)	8.41	8.44 (8.33–8.55)	I	8.37	8.22 (7.66–8.79)	7.65	7.63 (7.51–7.74)	I
	57.46	8.05	8.89 (8.19–9.60)	8.68	8.76 (8.64–8.87)	I	7.51	8.34 (7.76–8.92)	7.76	7.88 (7.76-8.00)	I
	74.84	8.42	8.31 (7.71–8.92)	8.49	8.54 (8.44–8.63)	I	7.53	7.71 (7.21–8.21)	7.58	7.61 (7.50–7.71)	I
	81.20	7.82	7.89 (7.27–8.50)	8.35	8.26 (8.16–8.35)	I	7.04	7.25 (6.74–7.75)	7.40	7.32 (7.21–7.42)	I
	88.23	7.35	7.27 (6.54-8.01)	7.88	7.82 (7.70–7.94)	I	6.75	6.59 (5.98–7.20)	6.97	6.88 (6.75-7.00)	I
	95.82	6.59	6.46 (5.43–7.48)	7.14	7.20 (7.04–7.37)	I	5.95	5.71 (4.86–6.55)	6.18	6.27 (6.09–6.44)	I

\*Yes, – no

(except in the mature wood at 50 °C and 30.54% RH), creating overlaps between their intervals of variation. This indicates that there are no significant differences between the methods.

No studies have compared the saturated salt method and DVS using the quadratic polynomials of the GAB model. However, this method can be considered valid, as the results obtained concur with those determined by comparing the *EMC* of DVS with the intervals of variation of the measurements using salts.

## 4 Conclusion

The isotherms of both juvenile and mature wood of *Pinus pinea* describe type II sigmoids and fit satisfactorily according to the GAB model. For any temperature and method, the juvenile wood has lower *EMC* than the mature wood.

The saturated salt and DVS methods show no significant differences when comparing the *EMC* points obtained with each one, both in the point-by-point comparison and using the quadratic polynomial form of the GAB model. This result is strengthened by the similar point of relative humidity from which multilayer sorption predominates over monolayer sorption in the two methods.

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#### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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