High Mechanical Performance Boards Made from Fibers of *Arundo donax* without Added Adhesives

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Arundo donax is a cane species with high growing productivity, and it is becoming an important source of biomass. The main objective of this study was to obtain fibreboards with high mechanical performance from *A. donax* without any added adhesive. Boards made without adhesive are free from formaldehyde emissions and consume no fossil resources. The characteristics of the obtained boards depended on the original material, steam explosion pre-treatments, and forming conditions (pressure, temperature, and pressing time). Production parameters were optimized. The effect of forming pressure on the physical and mechanical properties density, elastic modulus (MOE), modulus of rupture (MOR), tensile strength perpendicular to the faces (IB), thickness swelling, and water absorption of the obtained boards was studied. The European Norms (EN) methodologies were used to test the board specifications. Density, MOE, and MOR were modelled by a double reciprocal function. TS and WA were modelled with a reciprocal function in X. The boards obtained met and sometimes exceeded the requirements of these standards for the most demanding structural use.

Keywords: Arundo donax; Fibre Board; MOE; MOR; IB; TS; WA; Steam Explosion

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INTRODUCTION

Fiberboards can be produced without the addition of adhesives from different lignocellulosic materials (Boehm 1930; Mancera *et al*. 2008; Zhang *et al*. 2015; Pintiaux *et al.* 2015; Presenti *et al*. 2017). The characteristics of these boards vary depending on the raw material used, the production process, and the parameters used in the final assembly. These parameters are the conditions in the reactor pre-treatment, steam and forming conditions, which are recognized as the pressure of pressing (P_p), temperature of pressing (T_p), and time of pressing (t_p). Predicting the effects of these factors is essential to plan production operations in order to obtain the target characteristics of desired boards. The development of fiberboard with the addition of synthetic adhesives is well established and has produced good commercial results for years; however, this is not the case for producing high quality panels without adhesives. In recent years various plant species have been studied for the production of fiberboard, with the intent to define new materials of commercial interest to mitigate the environmental impact that comes from producing fiberboard. The ultimate goal is to increase the added value of forest, agricultural, and agribusiness products.

The applied pre-treatment has already been used for the preparation of biomass to build biopolymer-based materials (Focher *et al*. 1998; Anglès *et al.* 1999) and in preparing lignocellulosic materials for obtaining boards of other species besides *Arundo donax* (Ballesteros *et al*. 2000; Anglès *et al*. 2001; Salvadó *et al.* 2003; Quintana *et al.* 2009; Mancera *et al*. 2011; Mejía *et al.* 2014).

Pre-treatment conditions can be quantified using the severity factor proposed by Overend *et al.* (1987), where the effect of temperature and time in the hydrolytic pre-treatment is included. The physical and mechanical properties of the boards are affected by the severity of treatment.

Boards have been produced from many different materials including *Cynara carduncululus* (Mancera *et al*. 2008), *Miscanthus sinensis* (Velásquez *et al*. 2003), oil palm biomass (Hashim *et al*. 2012), and kenaf core (Xu *et al*. 2006). Reeds in general (and *A. donax* in particular) exhibit very high mechanical properties due to their structure and composition. Encouraging results obtained with *M. sinensis* have prompted the production of *A. donax* boards and investigation of their mechanical performance.

To facilitate industrial implementation, it may be important to know the relationship between the mechanical characteristics of the boards obtained and production parameters (pre-treatment and pressing conditions) applied. In this way mathematical models can be established that predict the optimal parameters for producing high quality fiberboards with predetermined mechanical characteristics.

A. donax is a large-sized reed (5 to 6 m high). The species is native to Asia and has been cultivated and traditionally used in different temperate zones as a horticultural guardian, cane screen, and to make musical wind instruments. Currently there is also interest in its potential as an energy source and source of pulp due to its rapid growth, ease of cultivation, and high yield of biomass per hectare.

A. donax boards should be studied to examine the convenience of using this species as raw material for the production of commercial fibreboard, or the possible prediction of the mechanical characteristics of such boards based on production factors. The objective of this study was to obtain boards of high performance and to model the effect of pressing pressure (P_p) on the physical and mechanical characteristics of *A. donax* fiberboards.

Recent studies have investigated the production of pulp and optimization of paper production using *A. donax* (Caparrós *et al*. 2007). Several studies have assessed *A. donax* as a potential energy feedstock (Szabó *et al.* 1996; Angelini *et al*. 2009; Mejdi *et al*. 2010). Other studies have investigated the effect of the stem morphology of *A. donax* on the characteristics of the pulp (Pereira and Shatalov 2002), its chemical composition (Monti *et al*. 2008), and the influence of solvents on the performance and pulp quality of this species (Shatalov and Pereira 2002). Other agronomic aspects, such as its propagation under different conditions (Ceotto and Di Candilo 2010) or the influence of fertilization on the yield (Monti and Zegada-Lizarazu 2017) and on the fuel quality of *A. donax* (Di Nasso *et al.* 2010) have also been assessed.

In the Mediterranean area, the spontaneous growth of *A. donax* in rivers and streams causes major problems with the evacuation of rainwater in avenues, reducing the flowing area of the channel and, when dragged, causing clogging in hydraulic works such as bridges and pipelines, thus increasing the risk of flooding. The use of *A. donax* as a raw material to produce fiberboards would aid management of this crop and would decrease the problems discussed above.

EXPERIMENTAL

Materials

The *A. donax* reeds used in this study were obtained from the banks of the natural course of a ravine in the municipality of Riudecanyes, a province of Tarragona, Spain. The rods used were two years old and were chopped with a GA 100 Black and Decker chipper (Towson, MD, USA), leaving pieces 3 to 8 cm long and about 2 cm thick. To prevent abnormal fermentation, the chopped material was stored in porous jute bags to reach equilibrium with environmental conditions until the completion of the pre-treatment.

Production Process to Obtain Fiberboards

Figure 1 shows the board production process using *A. donax*.

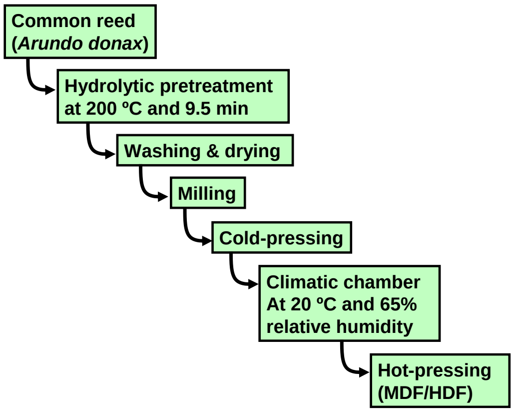

Fig. 1. Fiberboard production process

Steam Explosion

Chopped reeds, in batches of 800 to 1000 g, were introduced into a batch reactor composed of two parts: the reactor itself and an expansion chamber. Steam from a Borealis 380 V, 82 kW boiler at 42 bar (Vienna, Austria) was injected into the reactor to the required temperature (or pressure). Steam was then injected to reach 200 °C, and these conditions were held steady for 9.5 min. Then the valve connecting the reactor with the expansion chamber was opened, causing a sudden decompression. Disintegrated fibers were thus obtained. These fibers were covered with a highly reactive form of lignin, which facilitates the formation of boards *via* hot pressing, without the addition of exogenous adhesives.

The conditions (temperature and time) of steam explosion pre-treatment were defined previously on other fibers and similar materials such as *Miscanthus sinensis* (Velásquez 2002). Preliminary tests on *A. donax* were performed in order to obtain high-quality boards using BS EN 622-2: 2004 (2006).

Washing, Drying, and Milling of Pretreated Material

The exploded material was washed immediately after the steam explosion to remove degraded hemicelluloses that cause problems in the formation of boards. The washed material was spread in a drying chamber where it remained until coming to equilibrium with the environment, in a ventilated room at 25 ºC. Once a constant weight was achieved over several days, the material was stored until the milling stage. The remaining moisture of the material was about 12%, with little variation among sets.

Milling the exploded material produces positive effects on the characteristics of the obtained boards (Velásquez *et al.* 2002). Therefore, all dry pretreated material was ground prior to cold forming in a Restsch SM 100 mill (Düsseldorf, Germany) with a 4-mm sieve.

Cold Forming and Stabilization Until Constant Temperature (T) and Relative Humidity (RH)

The lack of geometrical uniformity of the boards is a fundamental problem in assessing data obtained from board products in scientific work. The findings on the effects of production parameters on the characteristics of the boards are not consistent or they even can be contradictory between different authors. In previous studies, mold filling with lignocellulosic material has been carried out directly on the hot press, and it is difficult to have a homogeneous distribution of the material in the mold. The first modification of the initial process consisted of making a cold pressing prior to making the board. This allowed the mold to be filled in a convenient way without limitation of time or risk of burns, leading to improved uniformity on the produced boards.

The solid obtained *via* steam explosion pre-treatment was washed, dried, ground, and then used as the starting point for next step. For each board 28.5 g of material was necessary to produce a board 150 x 50 mm and about 3 mm thick.

The weighted material was introduced and distributed evenly into a mold. It was cold pressed at 16 N/mm^2 in a conventional press (AN MEGA-30, Berriz, Spain), creating a pre-formed board. These boards were conditioned in a MMM Group Climacell 111 climatic chamber (Planegg, Germany) at 20 °C and 65% RH. Boards were kept in these conditions at constant weight.

Hot Pressing

Hot pressing was performed on a heated press (Servitec Polystat 300S, Wustermark, Germany). The pre-formed boards were placed in a mold on the bottom plate of the press. A steam evacuation mesh, a pre-formed board, a cover, and finally the mold piston were then placed inside the mold in the corresponding order. In the press the temperature, pressure, and pressing time (t_p) were set. The boards were pressed at 205 °C for 3.25 min, followed by a 1-min decompression (to allow the steam to get out of the test probe avoiding bubble formation), followed by a second pressing stage of 3.25 min. The total pressing time was 7.5 min.

Experimental Design

A one-way design with the single factor pressing pressure (P_p) at 6 levels (Table 2) was used. The dependent variables were the modulus of elasticity (MOE), modulus of rupture (MOR), internal bond (IB), density, thickness swelling (TS), and water absorption (WA). Several regression functions were fitted in order to determine the function that best described the trend of the effect. The statistical analysis was performed using Statgraphics Plus 5.1 software, (Statpoint Technologies, Inc., Warrenton, VA, USA).

Steam Explosion Board Characterization

The tested mechanical characteristics were selected considering the definition and classification of fiberboard established in UNE-EN 316: 2009 (2009). Provided that the hot pressing was performed using dry material in every sample, the standard applies to "boards made by dry process" (used for Medium Density Fiberboard, MDF). Among these, if the density is above 800 kg/m^3, they can be considered High Density Fiberboard, HDF.

Standard UNE-EN 622-5: 2010 (2010) sets the requirements of all fiberboard produced by dry process (MDF) for general and structural use in dry environments.

Table 1. EN Standard Tests for Physical and Mechanical Properties of Fiberboards and Values Required for Structural and General Purpose Boards

Board Type	Use	EN Standard	Test Method	Board Thickness (mm)	Checked Property	Value	Units
MDF	General	622-5	EN 317	2.5 - 4	TS	35	%
(HDF)			EN 319	2.5 - 4	IB	0.65	MPa
			EN 310	2.5 - 4	MOR	23	MPa
	Structural	622-5	EN 317	2.5 - 4	TS	35	%
			EN 319	2.5 - 4	IB	0.7	MPa
			EN 310	2.5 - 4	MOR	29	MPa
			EN 310	2.5 - 4	MOE	3000	MPa

The dimensions of the test pieces were chosen based on UNE-EN 325: 2012 (2012). For characterization of the boards the pieces were conditioned in the climate chamber at 20 ° C and 65% RH until a constant mass was obtained. Constant mass was obtained when the results of two consecutive weight measurements carried out at an interval of 24 h differed by less than 0.1% by mass.

The calculation of the MOE, MOR, and IB was made according to UNE-EN 310: 1994 (1994), UNE-EN 317: 1994 (1994), and UNE-EN 319: 1994 (1994) using a HOUNSFIELD H10KS universal testing machine (Salfords, UK).

To calculate the TS and WA of samples, specimens were immersed in a water bath for 24 h before the thickness was measured and the samples were weighed. The pieces were dipped into clean water in an upright position; the water had a pH of 7 ± 1 and a temperature of 20 ± 1 ºC. These conditions were maintained throughout the test. After 24 h the samples were pulled from the water, and excess water was removed. The samples were re-weighed and measured again. The scales used had a precision of 0.01 g (AND HM-120 scales, New York, NY, USA). The WA (defined as the percent absorbed water under these testing conditions) was determined relative to the weight of the original dry specimen. The thickness was measured using a digital micrometer (Mitutoyo 547-400S, Kawasaki, Japan).

RESULTS AND DISCUSSION

Results

The results of this study are presented in Table 2. The results were statistically analysed, and mathematical models were obtained to determine the effect of P_p in the studied parameters of the boards. Several mathematical models were checked for every parameter looking for a balance between model simplicity and good data fit. The most suitable model for each feature is presented.

Density

The density of the manufactured board ranged from 859.9 to 1295.3 kg/m^3. All boards were classified as high density (HDF) because they were above 800 kg/m^3.

Table 2. Production Parameters and Characteristics of Fiberboards made from *Arundo donax* at Various Conditions

Board Sample	Pp	MOE	MOR	IB	Density	TS	WA
	MPa	MPa	MPa	MPa	kg/m3	%	%
1	5	6525	38.26	2.16	117.32	14.97	25.00
2	5	5742	34.91	1.37	114.16	13.44	17.39
3	0.35	2239	11.16	1.42	859.90	18.46	58.24
4	5	5674	39.94	1.22	1157.65	8.71	20.22
5	5	6508	42.31	0.96	1179.86	9.57	22.22
6	5	7148	44.29	1.98	1217.46	10.47	18.89
7	5	6453	46.64	2.01	1224.09	8.81	18.68
8	9.6	6838	45.07	1.43	1216.03	9.69	14.61
9	5	5815	36.98	0.92	1168.61	9.18	24.44
10	5	6126	39.68	1.07	1174.25	13.76	22.73
11	10	7186	47.27	1.94	1146.91	11.32	20.65
12	12.5	8143	5.5	1.45	1034.60	9.55	16.13
13	15	8069	52.9	1.61	1130.12	11.71	18.60
14	15	9552	53.3	0.99	1174.16	10.03	15.73
15	12.5	8658	51.2	1.27	1155.20	12.30	15.22
16	12.5	6995	36.48	0.83	1295.27	8,.6	14.44
17	12.5	7028	36.8	0.90	1040.03	10.59	17.78
18	10	5510	37.5	0.92	1130.36	10.89	14.94
19	10	6037	35.7	1.02	1085.34	10.09	16.13
20	10	6465	42.39	1.42	1209.58	14.08	17.24

The density was modelled by a double reciprocal function of the form of Eq. 1, with a very low error probability (P-value < 0.0001),

$$\text{Density} = 1 /(a + b/P_\text{p}) \qquad (1)$$

where a and b are the parameters found in the fitting model.

The correlation coefficient was 0.8, showing a clear effect from the pressing pressure on density. The coefficient of determination was 64%. This indicates the effect of P_p in the density variability.

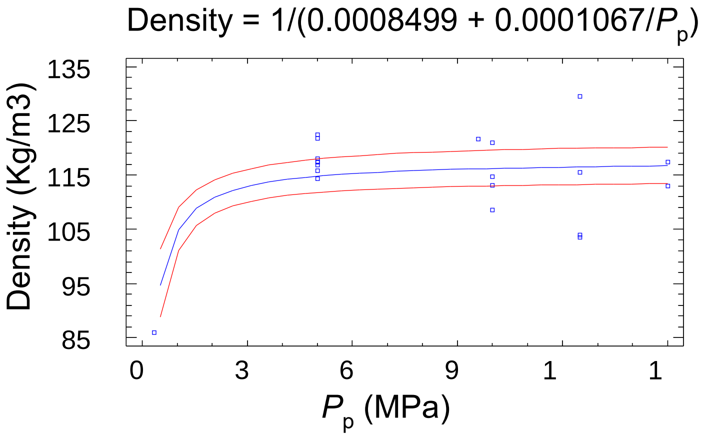

Fig. 2. Density dependence on P_p. External lines show a 95% confidence level interval

After increasing the pressing pressure, the board density was increased. At low P_p, a relatively low increase in pressure resulted in a significant increase in board density. At higher P_p the effect on the density was reduced. This is well explained with the chosen model, represented in Fig. 2. At higher pressing pressures density tended to an asymptotic value (1/a).

Modulus of Elasticity (MOE) in Flexion

MOE values in the analysed board samples ranged from 2239 to 9552 N/mm^2. All boards were over the 3000 N/mm^2 requirement from the EN standard for structural use, except sample No. 3, which was made at the lowest P_p (0.35 MPa).

As noted for density, a double reciprocal function of the form of Eq. 1 was the best model for the MOE/P_p relationship. The probability of error had the same value (P-value < 0.0001). Regression analysis gave a high correlation (0.967) and a coefficient of very high (93.5%) determination. Thus, pressing pressure had a clear and measurable effect on the MOE of *Arundo donax* boards (Fig. 3).

The MOE values obtained were very high, reaching 9552 N/mm^2. This value is far beyond what is required by UNE-EN 622-5: 2010 (2010) board standards as appropriate for structural use. It is of particular industrial interest that the model indicates from a Pp around 1 MPa that the minimum requirements of the standard for structural use boards were met.

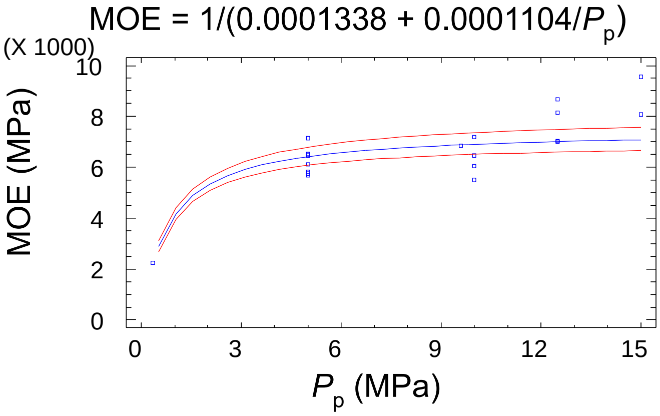

Fig. 3. Pressing pressure influence on MOE. External lines show a 95% confidence level interval.

Resistance to Flexion (MOR)

MOR values varied between 11.16 and 53.3 MPa. All boards except board No. 3 exceeded the 29 MPa required by UNE-EN 622-5: 2010 (2010) for structural use.

MOR dependence on pressing pressure was described by a double reciprocal function of the form of Eq. 1, and the model of this relationship had a very low error probability (P-value < 0.0001) (Fig. 4).

Both the value of the correlation coefficient (0.98) and the coefficient of determination (> 96%) were very high.

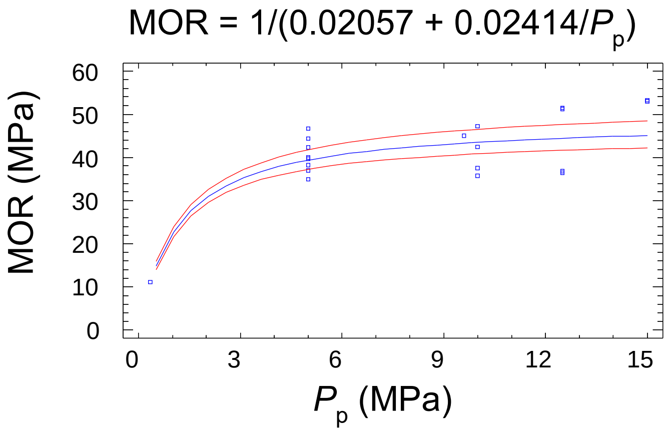

Fig. 4. MOR dependence on *P*p. External lines show a 95% confidence level interval.

Interestingly, the model indicated that from a P_p equal to or greater than around 3 MPa, the minimum requirements of the standard for structural use boards were met.

Internal Bond (IB)

IB values obtained varied between 0.83 MPa and 2.16 MPa. All values were above the 0.7 MPa required by UNE-EN 622-5: 2010 (2010) for structural use.

A clear relationship between IB values and P_p was not found. This is probably due to the fact that this test measures the strength of the weakest plane in the panel, making it more difficult to test statistical significance.

Thickness Swelling (TS)

TS values for the sample boards were between 8.1% and 18.5%, all well below 35%, which is the maximum allowed by UNE-EN 622-5: 2010 (2010) for structural use.

The proposed model is a reciprocal in X based on Eq. 2 and has a correlation coefficient of 0.66 and a coefficient of determination of 44% (Fig. 5),

$$\mathrm{TS} = a + b/P_p \tag{2}$$

where *a* and *b* are the parameters of the model. The correlation between Pp and TS was significant (> 60%) but lower than the correlation in MOR or MOE. The P-value was 0.0015.

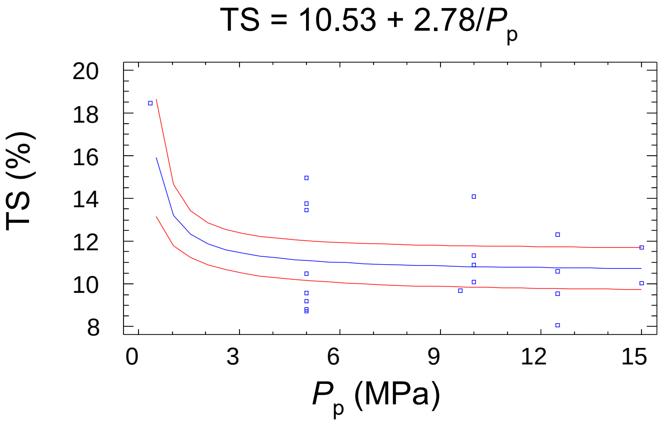

Fig. 5. TS dependence on Pp. External lines show a 95% confidence level interval.

Water Absorption (WA)

WA values ranged between 14.4% and 58%. All boards except No. 3 were below a WA of 30%.

The proposed model for P_p *versus* TS is a reciprocal in X according to Eq. 2. Regression analysis gave a correlation of 0.96 and a coefficient of determination of 92%. The results obtained and the model presented in Fig. 6 show the effect of pressing pressure on the water absorption of the boards.

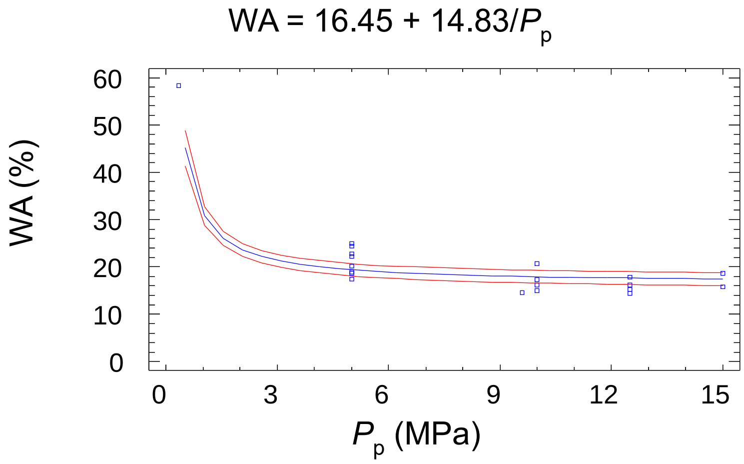

Fig. 6. WA dependence on Pp. External lines show a 95% confidence level interval.

CONCLUSIONS

1. *Arundo donax* L. is a suitable plant species for the production of fiberboard panels created by applying a steam explosion pre-treatment followed by a hot pressing. High mechanical performance boards can be obtained without the use of external adhesives.
2. The obtained panels have values of MOE, MOR, and (IB largely exceeding those required by standard UNE-EN 622-5: 2010 (2010) for structural use boards.
3. MOE and MOR were significantly influenced by pressing pressure (P_p). In all cases, a higher P_p lead to a higher MOE and MOR.
4. The effect of P_p on density, MOR, and MOE can be predicted with a simple model with high level of accuracy. The obtained regressions can help optimizing the production costs of fiberboard panels. The relationships between these properties have been modelled with a model of reciprocal double adjustment.
5. The TS and WA values also had a high dependence on P_p. In all cases, increasing P_p resulted in boards with lower TS and WA values. The TS values observed also largely exceed the requirements in the UNE-EN 622-5: 2010 (2010) standards for structural use boards. For TS and WA, a reciprocal model in X (Eq. 2) was the best fit for the dependence of those properties on P_p. The obtained lines of regression can help optimize the production costs of fiberboard panels.

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