Investigation of Alkaline Surface Treatment Effected on Flax Fibre Woven Fabric with Biodegradable Polymer Based on Mechanical Properties

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Highlights:

- Composites were fabricated using thin film casting and hot-pressing.
- The effects of alkaline treatment on the flexural and impact properties of flax fibre reinforced polylactic acid (PLA) thin film were investigated.
- Optical images of the treated flax fibres also showed improvement.

Abstract. Biodegradable polymers such as polylactic acid (PLA) are used nowadays due to their degradability, durability and environmentally friendly properties. Alkaline surface treatment of natural fibres is used to increase the flexural properties of composites. This research investigated the flexural properties of dry compressed PLA, wet mix PLA, untreated flax/PLA and treated flax/PLA composites. The flax fibre was first treated with NaOH. The wet mix PLA was prepared via solvent casting with chloroform and dried at room temperature overnight followed by post-drying in an oven. The flax/PLA composites were fabricated using a hot press for 6 minutes. The wet mix PLA showed higher flexural strength compared to the dry compressed PLA. The treated flax fibre composite showed higher flexural strength compared to the untreated flax fibre. The flexural strength and elongation at break of the treated flax fibre composite was increased by 4.79% and 27.76%, respectively, while the flexural modulus decreased by 0.79% compared with the untreated flax composite. The treated flax composite also showed an improvement in impact properties, increasing its impact strength by about 3% and 10% at energy levels of 10 J, 15 J, and 17.5 J compared with the untreated flax fibre. Therefore, the investigation of the surface treatment of flax in a PLA matrix based on its mechanical properties revealed better properties compared to untreated flax/PLA composite.

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1 Introduction

Several applications that use composites reinforced by natural fibres have attracted the attention of the composites community in the current year. Due to synthetic fibres requiring high expenses, natural fibres have been studied as potential reinforcement in composites applications [1,2]. The properties of natural fibre composites have been considered for a wide range of natural fibres, including flax, jute, hemp, banana, bamboo, pineapple, agave and rubber wood in several different studies. In order to achieve better reinforcement of polymer matrix composites it is important to relate their characteristics to the properties of the natural fibre, such as low density, low cost and biodegrability [3,4].

There are many different types of natural fibres with good mechanical properties, one of which is flax fibre. Murali [5] found that the specific tensile strength of flax fibre is comparable to that of glass fibre. Many forms and configurations of flax fibre are used in applications such as mats, roving, fabrics and monofilaments. There is a variety of manufacturing techniques to produce these forms of fibres. Using flax fibres normally has relatively low cost compared to using glass fibres. There are negative effects of using glass fibres, as reported by Bos [6], where these are suspected of causing lung cancer, whereas there is no such problem with natural fibres. The process of burning flax fibres with few slag left gives the thermal recycling of flax fibres a great advantage over glass fibres. Thus, it makes flax a suitable fibre to replace conventional glass fibre as reinforcement of composite materials [7-10].

A natural fibre like flax has unique properties that are fundamentally different from glass and other synthetic fibres. As a known natural fibre, flax is majorly influenced by its hydrophilicity. This characteristic leads to poor matrix adhesion and high water absorption due to the presence of free hydroxyl and polar groups in the fibre itself [11,12]. To improve the interface properties and adhesion to the polymer matrix, a number of different chemical treatments can be used, such as alkaline treatment, silane treatment, benzoylation, acetylation and enzymatic treatment [13-16]. Waxy substances are chemical substances in the fibre. The ratio of cellulose will be increased by the removal of these substances by chemical treatment of the fibre. The mechanical properties of the fibre are improved while the water absorption is decreasesd by using these treatments [11,13]. Huner found that sodium hydroxide (NaOH) treatment improved the mechanical properties of an epoxy matrix composite [17]. Van de Weyenberg found that NaOH has high swelling compared to potassium hydroxide (KOH) or lithium hydroxide (LiOH) [18]. Alkali treatment can give an increase of up to 30% in the longitudinal

properties due to the removal of pectins [19]. Thomas concluded that tensile testing of the fibres showed improvement in strength by increasing the soaking time duration up to 2 hours.

Polylactic acid (PLA) is derived from 100% renewable resources of a naturally occurring organic acid, i.e. lactic acid. PLA is well known as a biodegradable, highly versatile aliphatic polyester [18]. PLA melts at low temperature, in the range of 180 to 220 °C, with a glass transition temperature of 60 to 65°C. Moreover, the formation of non-harmful and non-toxic compounds from PLA and its composites results from simple hydrolysis of the ester backbones, which can degrade easily, making PLA degradable in nature. Recycling of PLA to its monomer can be done by using hydrolysis or thermal depolymerisation. PLA also provides easy access to a wide range of materials that can easily be processed by injection moulding, casting, extrusion and spinning. Thus, PLA is an ideal polymeric material for different practical applications all over the world based on its biocompatibility, complete biodegradability and the non-toxic nature of its degradation products [20].

In this study, two types of PLA composite were investigated, including two types of flax fibre: treated with alkaline and untreated. The two types of PLA used were pellets and thin film layers. Both types of PLA were tested in neat form without any reinforcement for flexural analysis. Laminates of treated and untreated flax fibre were fabricated with the PLA thin film composite. The prepared composites of both types of flax fibre were subjected to low-velocity impact testing with a drop weight machine. The effects of alkaline treatment on the flax fibre were then observed with an optical microscope.

2 Materials and Experiments

2.1 Materials

In this study, woven flax fibre was used as received from Mecha Solve Engineering. The polylactide acid thermoplastic was purchased from Innovative Pultrusion Sdn. Bhd. NaoH was used to treat the flax fibre and chloroform was used as solvent. Both chemicals were purchased from R&M Chemicals.

2.2 Composites Production

2.2.1 Fibre Surface Treatment

Woven flax fibre was used as reinforcement, while PLA was used as the matrix in the composite. The woven flax fibre material was cut into dimensions of 260 x 260 mm per piece as mould size. The fibre then underwent surface treatment

by using alkaline treatment via an NaOH solution. The fibre was treated by soaking in a 2% NaOH solution for 2 hours.

After treatment, the fibre**s** were washed with distilled water until the pH became natural. Then, the fibre**s** were dried at room temperature for 48 hours. The dried fibres underwent further drying in an oven at temperature 80 °C for 24 hours to remove excess water.

Figure 1 Example of the composite lay-up.

2.2.2 Fabrication of Composite

The wet mix PLA was cast by mixing PLA in pellets with chloroform at a ratio of 1:5. The solution was then stirred using a mechanical stirrer for 30 minutes with a speed of 500-700 rpm. For the first 15 minutes, heat at 60 ℃ was applied to the solution. After 30 minutes of stirring, the solution was poured into a steel mould and dried in an oven at 55 ℃ for 2 hours and 30 minutes. On the other hand, the dry compressed PLA without chloroform was prepared by hot pressing of the PLA in pellet form.

Laminate fabrication of flax with the wet mix PLA composite was done using the hot press method. The flax fibre, wet mix PLA and dry compressed PLA were pressed by using a manual hot press machine at 20 tonnes pressure and a temperature of 190 ℃. The laminate specimens consisted of six layers of wet mix PLA and five layers of flax in the middle, as shown in Figure 1. After 5 minutes of pre-heating, hot-pressing was done for 1 minute with 5 times bumping. Mild steel plates with dimensions of 260 x 260 x 32 mm were used in the hot-pressing process as the mould. The dry press PLA, wet mix PLA, untreated flax/PLA and treated flax/PLA composite are herein represented by 'P', 'T', 'UF' and 'TF'.

2.3 Characterizations

2.3.1 Flexural Test

The flexural properties of the composites were determined using three-point flexural bending test according to the ASTM D-790 standard using a Universal

Testing Machine (Instron, Model 3365). Ten specimens were tested for each sample at room temperature with a crosshead speed of 2.0 mm/min and a gauge length (support span) of 60 mm. The specimens had a rectangular shape of 125 x 12.7 x 3.2 mm per piece.

2.3.2 Low Velocity Impact Test

The impact strength of the composites was determined by using a low-velocity impact machine (Imatek Ltd, type 8000D, model D5000, Knebworth, UK), also known as a drop test machine. The drop test was performed according to the ASTM D7136 standard, with dimensions of 150 x 100 x 3.2 mm per piece. Three specimens were tested for each sample of untreated flax and treated flax for three different energy loadings, i.e. 10 J, 15 J, and 17 J. The impact energy of the specimens was tested and calculated according to Eq. (1):

$$F=mgh \tag{1}$$

where F is the force (kN), m is mass (g), g is gravity (m/s), and h is the height (m) of the striker before release to impact the specimen, depending on the value of the impact energy as stated in Table 1.

Table 1 Value of impact energy and striker height.

Impact Energy (J)	Height of Striker (m)
10	0.2
15	0.3
17.5	0.35

2.3.3 Morphology Analysis

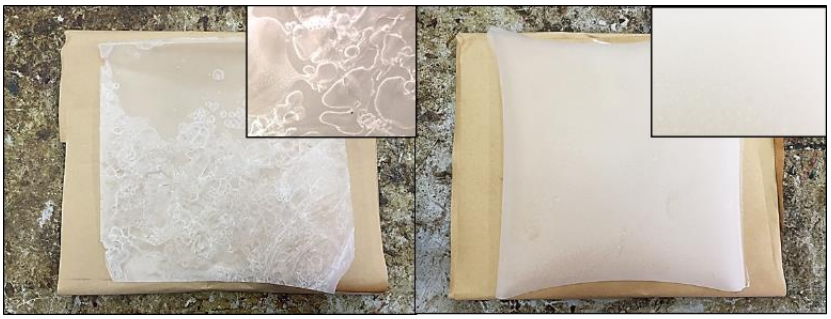
Changes in the surface morphology of the treated and untreated flax fibre were observed under an optical microscope using an Olympus Optical Microscope (OM). The image surface was captured at 50x magnification.

3 Results and Discussion

3.1 Observations on PLA Thin Film Casting Method

Initially, different drying temperatures were investigated to identify PLA thin film that produced low trapped bubbles. The thin film of wet mix PLA in Figure 2(a) was dried in an oven at a temperature of 80 °C. The surface produced many bubbles after drying. The thin film in Figure 2(b) was dried in an oven at a temperature of 55 °C, producing a smooth surface and easier handling. As reported by Mohan, bubbles form during the first thirty minutes of drying due to the large amount of chloroform trapped in the sample when the solidified casting

is done too fast [21]. Therefore, a low drying temperature of 55 °C was used to slow down the solidification process and mitigate bubble formation.



(a) (b)

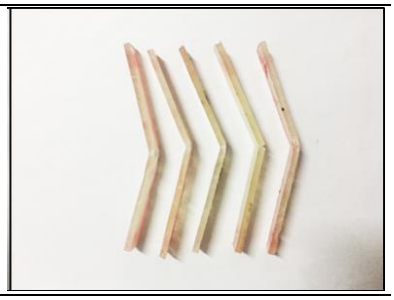
Figure 2 PLA thin film after drying at different temperatures: (a) 80 °C, (b) 55 °C.

3.2 Flexural Properties of the Composites

A flexural mechanical test was performed up to initial cracking until the failure of the matrix of the polymer composite to analyse the elastic behavior of the fibre and the matrix. Table 2 shows selected samples of the dry press PLA, wet mix PLA, untreated flax/PLA and treated flax/PLA composites after flexural testing. It can be clearly seen that the dry compressed PLA samples were broken, while the wet mix PLA was only bent at certain angles. The untreated flax/PLA composite was clearly delaminated before failure, which indicates early failure due to the limited bending angle when load is applied to the composite. However, the treated/PLA composite bent only slightly. Figure 3 shows the flexural strength of the dry compressed PLA, wet mix PLA, untreated flax and treated flax under loading. A great difference was recorded for the flexural properties, where the wet mix PLA and TF had much greater values than the dry press PLA and UF. From the results, the flexural strength of the wet mix PLA increased by 9% compared to the dry press PLA in pellet form. The NaOH surface treatment of the flax fibre improved the flexural strength, i.e. an increase of 11 MPa or 5% compared to the untreated flax composite.

The elongation at break of the treated flax is shown in Figure 5, which shows the flexural extension properties of all composites. The tendency to delaminate of composites that contain flax fibres may strongly depend on the stacking sequence selected, as observed in [22]. Therefore, the configuration presented is not specific, while the results are not definite.

Table 2 PLA Composites after flexural testing of dry press PLA, wet mix PLA, untreated flax/PLA and treated flax/PLA composites.

Sample	PLA in pellet form	PLA in thin film form
Polylactic acid (PLA)		
Sample	**Untreated flax fibre**	**Treated flax fibre**
Flax/PLA composite		

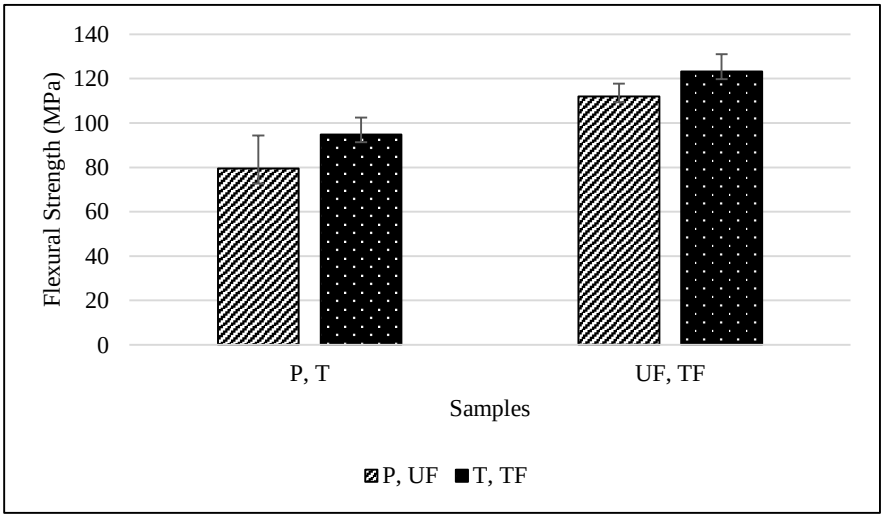


Figure 3 Flexural strength of different PLA process and flax/PLA composites.

The flexural modulus showed a similar trend as shown in Figure 4. The flexural modulus of the wet mix PLA and the treated flax/PLA composite were reduced

slightly, by 0.32 and 0.09 GPa, i.e. with 4.18% and 0.79%, respectively. The decrease in flexural modulus may be related to the high strain ratio of the wet mix PLA and the alkaline surface treatment, which affects their flexural behavior. Polymer materials generally have no regular structure in the polymer chains, which are loose. Therefore, plastics have high ductility. For example, when force is applied to them, they are able to stretch out due to the amorphous phase when being composed. This can be compared to the PLA thin film results. Liu states that lamellae crystals will form during polymer crystallization when the polymer chains fold into layers and stacking. A rigid structure is created during this process [23].

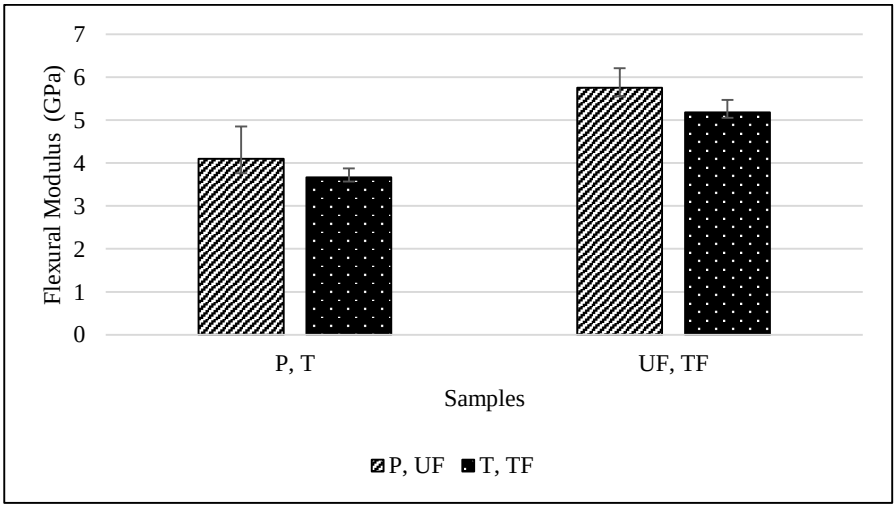


Figure 4 Flexural modulus of different PLA process and flax/PLA composites.

Figure 5 shows higher flexural strain displayed by the wet mix PLA, with an increase by 10% and about 50% compared to the dry press PLA, while the treated flax/PLA composite also showed an improvement by 2% and about 20% compared to the untreated flax/PLA composite. The alkaline treatment improved and enhanced the flexural strength and elongation at break as revealed by the flexural mechanical testing results compared to the untreated flax/PLA composite, as shown in Figures 3 and 5.

This enhancement of the flexural properties can be explained by better adhesion of the fibres after the alkaline surface treatment, which removes waxy substances from the fibres [11]. As reported by Yan, the alkaline treatment of flax composite increased the flexural strength by 16.1%, respectively, while in this study the flexural strength was increased by 5%. The removal of impurities from the fibre surface enhances the flexural properties due to the increased amount of cellulose.

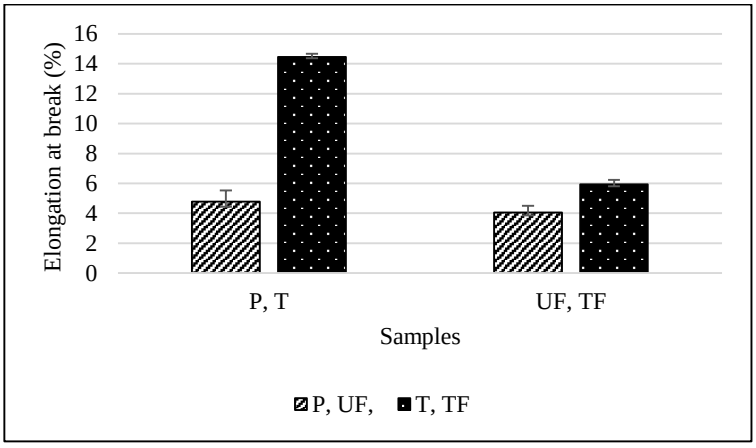
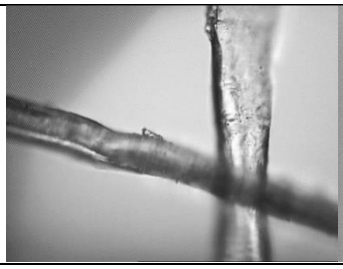
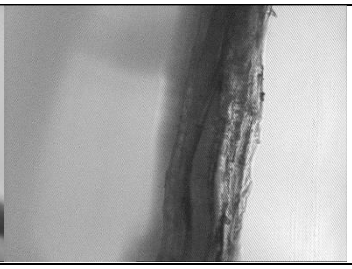


Figure 5 Flexural modulus of different PLA process and flax/PLA composites.

Table 3 shows optical images of the untreated and treated flax fibres. The treated flax fibres had a rough surface, while the untreated flax fibre surface was smoother. This is due to the result of chemical treatment using NaOH, which makes the fibre surface rougher and creates physical interlocking with the polymer [24]. Shibata has reported that fibrillation improves the interfacial region and that the contact surface area with the matrix is more effective. According to Reddy, the inner fibre structure, stiffness and density decreased after the removal of wax, lignin and hemicellulose from the fibres after NaOH surface treatment. The rough surface of the treated flax fibres increases the adhesion between the matrix and the fibres.

Table 3 Optical images of untreated flax fibre and treated flax fibre at 50x magnification.

Magnification	Untreated flax fibre	Treated flax fibre
50X		

Fewer gaps in the interfacial region lead to better adhesion between the matrix and the fibres. Thus, the area of the fibres for the wetting process is also increased. After treatment with NaOH, the fibre bundle become loose due partial removal

of some of the lignin during the treatment. In addition, more wetting areas are exposed on the fibre surface to create a good contact with the matrix [25]. On top of that, the fibre structure is disrupted, hence reducing the void content in the fibres to reveal a compressed cellular structure after the alkaline surface treatment [26].

Figure 6 shows the impact properties based on the strength of the untreated/PLA and the treated flax/PLA composites at impact energy levels of 10 J, 15 J, and 17.5 J. As shown in Figure 6, there is a similar trend of increased impact strength of the UF and TF composites for all energy levels.

At an energy level of 10 J and 15 J, the TF impact strength increased with 0.63 J and 0.86 J, or 3.2% and 3% for both the UF and TF composites, while at 17.5 J the increase was about 3.22 J, or 11% compared to the untreated flax.

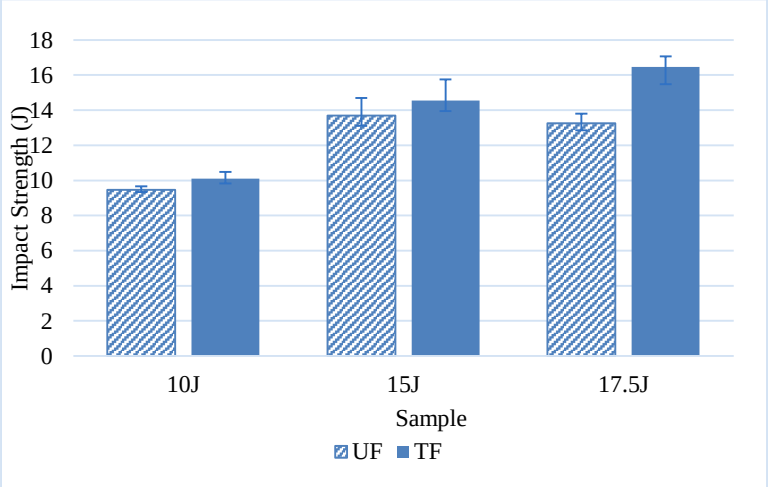


Figure 6 Impact properties of untreated and treated flax fibre based on strength with different energy loadings.

Table 4 provides more information about the impact test at three different energy levels (10 J, 15 J, and 17.5 J) for the untreated flax/PLA and the treated flax/PLA composites. The maximum force of UF and TF is slightly different, where TF had higher force compared to UF for the composite impacted at energy 15 J and 17.5 J. However, at an impact energy of 10 J the force of TF was lower than that of UF. As for the impact energy, UF impacted from a height of 0.3 and 0.35 m resulted in penetration of the flax fibre laminates, which did not occur with the TF laminates. This shows that the alkaline treatment improved the impact properties of the flax fibre.

Table 4 Average maximum load (kN) and non-elastic energy during impact at different energy levels.

Energy (J)	Laminate	Max load (kN)	Absorbed energy (J)	Max displacement (mm)
10	UF	1.105	9.465	13.275
	TF	0.965	10.095	14.075
15	UF	0.943	13.68667	28.42667
	TF	1.5	14.55	17.29
17.5	UF	0.94	13.25	36.47
	TF	1.26	16.47	17.85

Table 5 Image of untreated and treated flax fibre after low velocity impact at different energy levels.

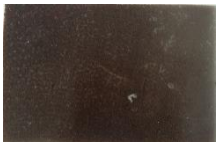
Energy (J)	View	Untreated Flax fibre	Treated Flax fibre
10	Front		
	Back		
15	Front		
	Back		
17.5	Front		
	Back		

Table 5 shows a front and back view of the untreated and the treated flax fibres after low-velocity impact at energy levels of 10 J, 15 J, and 17.5 J. Different lamination occurred due to the effect of the impact, producing damage in the composite. Extensive tearing from the impact energy was noticed in all the untreated flax composites. A comparison of all impact energy levels shows that the tearing of UF was more extensive compared to that of TF.

The extension of crack tearing increased with increasing the impact energy. For an impact energy level of 10 J the composites did not tend to be fractured away from the TF composite, while at 15 J UF the composites were broken. Increasing the impact energy to 17.5 J, the UF crack tearing extended until the end of the composite and the sample was damaged. The entire sample showed pine-tree cracking damage, where the crack propagation became larger at the back of the impacted area compared to the impacted area.

4 Conclusion

In this study, the effects on the flexural and impact properties of dry press PLA, wet mix PLA and flax/PLA composites were investigated and evaluated. The results showed that the wet mix PLA and treated flax/PLA composites had higher flexural strength and elongation at break compared to the dry press PLA and untreated flax/PLA composites. Optical images of the treated flax fibres showed an improvement of the surface condition compared to the untreated fibres. The impact strength for the treated flax/PLA composite was improved, where the strength of the laminates increased, especially during impact at energy levels of 15 J and 17.5 J. Therefore, combining wet mix PLA and treated flax woven fibres produces better mechanical properties.

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