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Advanced hemp-based hybrid composites: A study on the influence of various nanofillers on mechanical and morphological properties

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Abstract

This study investigates the mechanical and microstructural attributes of epoxy composites with hemp fiber reinforcements and incorporated aluminum oxide (Al_2O_3), silicon dioxide (SiO_2), and magnesium oxide (MgO) nanoparticles at different weight fractions of 0.5 wt%, 1.0 wt%, and 1.5 wt%. Nano-fillers remarkably improved the composite performance, as indicated by tensile and flexural tests conducted by ASTM D3039 and ASTM D790 standards. The composite containing 1.5 wt% Al_2O_3 outperformed other nano filled composites with a tensile strength of 64 MPa and tensile modulus of 3.6 GPa, versus 52 MPa and 3.0 GPa for the unfilled hemp/epoxy composite. In parallel, flexural strength and modulus also improved from 78 MPa and 3.5 GPa (unfilled) to 93 MPa and 4.2 GPa with 1.5 wt% Al_2O_3. SEM images corroborated the improved interfacial bonding and diminished fiber pull-out in nano-filled composites. The addition of nanofillers, mainly Al_2O_3, greatly improved the structural integrity of the hemp-based green composites, allowing for their use in sustainable load-bearing engineering applications.

1. Introduction

As science and technology advance at an alarming rate, industries like manufacturing are shifting to cleaner and more environmentally friendly economic growth. As substitutes for conventional resources, nano-biocomposite (NBC) substances have surfaced with promising properties and capabilities for a range of applications in many industries (aviation, automobiles, furnishings, packaging, transit, healthcare, and defence industries [1]. One of these elements is in matrix form, while the others are in particulate or fiber format. Any load that is placed on NBC components is distributed equally across all of the components. Improved productivity in industries, simplicity of manufacturing gadgets, and lower production costs are the main advantages of polymer-based NBC materials. Fiber-reinforced composites, particularly natural fiber–reinforced biocomposites (BCs) and nano-biocomposites (NBCs), offer several advantages over conventional synthetic composites [2]. Combining inorganic or organic polymers and nanoparticles with natural sources offers a chance of enhancing mechanical properties and hence broadening the range of applications. Because of their ability to degrade, inorganic nanoparticles are currently being studied for incorporation into NBCs by combining them with natural fiber in the matrices. In a composite system, leads to the formation of an interface link among the natural fibers and polymers, while the organic stage aids in the formation of the inorganic matrices [3,4].

Due to their low cost, minimal environmental impact, and mechanical properties comparable to those of petroleum-based synthetic fibers, natural fibers such as oat, hemp, jute, and cotton are considered viable reinforcement materials for natural fiber-reinforced polymers (NFRPs). Among these, industrial hemp (Cannabis sativa L.) is an environmentally sustainable fiber crop owing to its rapid growth rate, high biomass yield, and adaptability to a wide range of cultivation conditions. Furthermore, hemp is regarded as a renewable resource with significant potential for use in sustainable composite applications [5]. The stalks are used to make hemp fibers (HFs), which are subsequently transformed into fibers by retting, breaking, scutching, and hackling. Fibers are widely utilized in many different industries, such as textiles, paper, composite materials, and more. They are composed of a bast and a core layer called a hurd. Furthermore, hemp fibers are high in cellulose (70% to 72%), hemicellulose (19%), lignin (5%), and additional components [6]. Compared with other natural fibers, hemp exhibits a high aspect (dimension) ratio and a relatively high Young’s modulus, making it an attractive reinforcement for polymer composites. However, practical limitations arise from poor interfacial compatibility between hydrophilic hemp fibers and polymer matrices, leading to reduced mechanical performance, increased strain, and moisture sensitivity. These issues are clear indications of weak interfacial adhesion within the composite system. Such interfacial deficiencies result in inefficient stress transfer, reduced dimensional stability, and premature failure through fiber–

matrix debonding and delamination [7,8]. Owing to these compatibility issues, only specific thermosetting matrices, such as epoxy and polyurethane, are commonly suitable for hemp fiber reinforcement. To address these challenges, interface modification through surface treatments and the incorporation of nanomaterials has emerged as an effective approach [9]. In addition to enhancing interfacial mechanical bonding, nanofillers promote mechanical interlocking, improve stress transfer across the interface, and contribute to crack deflection and energy absorption mechanisms. Consequently, nano-enabled composite systems exhibit improved durability and overall mechanical performance, highlighting the need for interface-engineered composite designs [10].

However, for many years, engineering applications of hemp fiber materials have been constrained by the fibers' toughness and rigidity. Consequently, in addition to hybridizing with other high-modulus and high-strength filaments, this research also attempts to investigate novel approaches to address this issue [11]. In this context, nanofillers are now a dependable option to improve the mechanical properties of hemp fiber materials even more; no study findings have ever been published in this area. Three different kinds of nanofiller/epoxy composites were made in this work using the compression molding technique. To evaluate their effect on fortifying the ductile epoxy resin matrix, their mechanical and morphological characteristics were examined. Then, using compression molding techniques, nano-filler/hemp fiber/epoxy hybrid composites were also made, and their mechanical attributes were examined and contrasted. Lastly, scanning electron microscopy observations were used to examine the fracturing patterns in nanofiller/epoxy hybrids and nanofiller/hemp fiber/epoxy composites. This work might advance the investigation and use of hemp fiber materials and provide a foundation for inexpensive structural composite items by elucidating the reinforcement impact of nano-filled materials on epoxy resin.

2. Experimental works

2.1 Materials

This study utilized 300 GSM plain-woven hemp fabrics, which were obtained from the Natural Fiber Industry located in Madurai, Tamil Nadu, India. The matrix was composed of epoxy resin E-51, and the curing agent was polyether amine D-400, also acquired from GVR Chemicals located in Madurai, Tamil Nadu, India. The filler nanoparticles—magnesium oxide (MgO), silicon dioxide (SiO_2), and aluminum oxide (Al_2O_3)—were purchased from Deekshi Scientific Solutions in Bangalore, Karnataka, India. Each had an approximate particle size of 30 nm. The seller provided phase information, which indicated that the MgO was cubic, SiO_2 was amorphous, and Al_2O_3 was in the γ phase. Reinforcement and filler materials are shown in the photographs in Figure 1.

2.2 Chemical treatment

For 4 h at room temperature, the hemp fibers were treated with 5 wt% concentrations of NaOH. The treated fibers were dried in the sun to eliminate moisture and washed multiple times with mineral-rich water to get rid of any remaining NaOH from the fiber surfaces. After removing any remaining moisture with natural sunshine, they were wrapped in plastic wrap and kept. Following treatment, the hemp was put in a dry box with 40% humidity. Equation (1) shows the response that the therapy produced [12].

$$\text{Fiber-OH} + \text{NaOH} \rightarrow \text{Fiber-O-Na} + H_2O \qquad (1)$$

2.3 Composite fabrication

The fabrication of epoxy/hemp/nanofiller hybrid composites utilized the hand layup technique, which was then followed by compression molding. The initial stage consisted of combining epoxy resin and nanofillers in varying load weights of 0.5 wt%, 1 wt%, and 1.5 wt%. The initial stage of the process was carried out by mechanically stirring at a rate of 1200 rpm for 10 h, followed by ultrasonic dispersion for 2 h targeting the breakdown of agglomerates to improve the distribution of nanoparticles in the matrix. Afterward, the hardener was added to the resin and the mixture was stirred consistently for 5 min. The resin–nanofiller mixture was then applied to six layers of the hemp fabric through the hand layup method to achieve hemp fabric impregnation. The impregnated laminates were placed into a compression mold (300 mm × 300 mm × 3 mm) at 100°C and 4 MPa of pressure for 180 min. The composites were demolded, and the ASTM standard specimen dimensions were achieved using a laser machine. Table 1 contains details regarding the composites, while the overall sequence of fabrication is illustrated in Figure 2.

2.4 Composite characterizations

The hybrid materials were formed into a rectangular form and subjected to a tensile test under ASTM D-3039-76 (250 mm × 25 mm × 3 mm). Automated universal testing equipment was used to perform tensile tests at a loading rate of 2 $mm \cdot min^{-1}$. Comparable to flexural testing (127 mm × 12.7 mm × 3 mm) carried out by ASTM D-790. The test equipment has been detailed above, and these tests were performed in a three-point bending mode with a loading rate of 2 $mm \cdot min^{-1}$ and a span measurement of 18 mm [13].

Figure 1. Photographic images of (a) Hemp fiber mat; (b) Al_2O_3 NPs; (c) SiO_2 NPs; (d) MgO NPs and (e) Matrix materials.

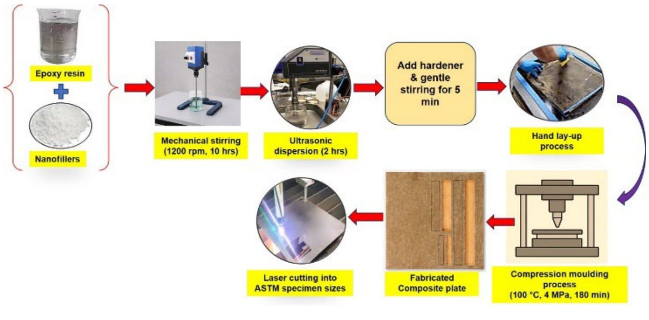

Figure 2. Graphical representation of hybrid composite fabrication process

Table 1. Fabricated composites concerning various nano filler additions.

Sl. No	Symbols	Composition	Al_2O_3 [wt%]	SiO_2 [wt%]	MgO [wt%]
1	A	Plain epoxy	0	0	0
2	B	Epoxy/hemp	0	0	0
3	C	Epoxy/hemp	0.5	0	0
4	D	Epoxy/hemp	1	0	0
5	E	Epoxy/hemp	1.5	0	0
6	G	Epoxy/hemp	0	0.5	0
7	H	Epoxy/hemp	0	1	0
8	I	Epoxy/hemp	0	1.5	0
9	J	Epoxy/hemp	0	0	0.5
10	K	Epoxy/hemp	0	0	1
11	L	Epoxy/hemp	0	0	1.5

2.5 Microstructural analysis

Both before and after the mechanical testing, the material's microstructure and fracturing structure were examined using scanning electron microscopy at an acceleration voltage of 10 kV. Before assessments, the specimens were gold-plated and sputtered for one minute. All the mechanical testing and microstructural analysis were also performed at SRM University, Chennai, Tamil Nadu, India.

3. Result and discussion

3.1 Tensile properties

The enhancement of tensile performance in the fabricated hemp fiber-reinforced epoxy composites was analyzed to determine the reinforcing influence of Al_2O_3, SiO_2, and MgO nanoparticles. Without fiber reinforcement or nano-fillers, the epoxy resin had the lowest value of 35 MPa for tensile strength and 2.3 GPa for tensile modulus, as observed in Sample A. Incorporating NaOH-treated hemp fibers into the epoxy matrix resulted in Sample B having a tensile strength of 52 MPa and a tensile modulus of 3.0 GPa. This improvement can be attributed to the inherent mechanical strength and load-bearing capability of the hemp fibers, along with enhanced mechanical interlocking at the fiber–matrix interface resulting from the NaOH treatment. The NaOH treatment facilitates the removal of surface impurities from hemp fibers, such as waxes, pectin, hemicellulose, and lignin, resulting in a cleaner and rougher fiber surface. As a consequence of this surface modification, a greater number of active hydroxyl groups become exposed, and micro-scale cavities, grooves, and pits are formed on the fiber surface [14]. When these treated fibers are embedded in the epoxy matrix, the surface irregularities allow the resin to penetrate into the fiber structure, thereby physically anchoring the epoxy and enhancing mechanical interlocking at the fiber–matrix interface [15]. Consequently, improved interfacial adhesion enables more efficient stress transfer from the epoxy matrix to the hemp fibers under tensile loading, which explains the observed increase in tensile strength and tensile modulus for the composites fabricated with NaOH-treated hemp fibers (Sample B). Adding Al_2O_3 NPs to the hemp/epoxy composite system bolstered tensile strength across all samples [16]. The sample C, with 0.5 wt% Al_2O_3, had 56 MPa of tensile strength and 3.2 GPa tensile modulus. Increasing the proportion of Al_2O_3 to 1.0 wt% and 1.5 wt% in samples D and E, respectively, improved the values of tensile strength to 60 MPa and 64 MPa and the modulus to 3.4 GPa and 3.6 GPa. The three reasons for this trend are the high mechanical stiffness and hardness and the nanoscale size of Al_2O_3, which enhances stress transfer efficiency between the matrix and fibers. Also, well-dispersed nanoparticles can mitigate crack propagation, increasing overall strength and rigidity. Figure 3 shows the tensile strength and modulus of hybrid composites [17].

For SiO_2-filled composites (Samples G–I), an analogous increasing trend in tensile characteristics was noted, albeit lower than for Al_2O_3-based systems. Sample G exhibited a tensile strength of 54 MPa and a modulus of 3.1 GPa, which for Sample I increased to 62 MPa and 3.5 GPa, respectively, at 1.5 wt% loading. This increase was attributed to the SiO_2 concentration. Added SiO_2 nanoparticles serve as efficient fillers for the microvoids due to their high surface area and chemical stability, thereby enhancing interfacial adhesion. It could be proposed that the modest decrease in performance could be due to the relatively lower intrinsic stiffness of SiO_2 when contrasted with Al_2O_3. Still, the improvement is remarkable considering the neat hemp/epoxy composite, which indicates that SiO_2 does enhance load-bearing capacity. The tensile behavior of the MgO-based composites (Samples J–L) was also improved but was lower than the Al_2O_3 and SiO_2-reinforced systems [18]. Sample J exhibited a tensile strength of 53 MPa and a modulus of 3.0 GPa, which improved to 59 MPa and 3.2 GPa at 1.5 wt% for Sample L. There is a possibility offered by MgO in considering it as a reasonable reinforcement; however, its lower modulus and poor interfacial bonding characteristics with the epoxy may reduce the strengthening effect when compared to Al_2O_3 and SiO_2. However, it is effective to some extent in further enhancing the matrix morphology and additional mechanical properties owing to filler-matrix interaction and possible mechanisms of plastic deformation under tensile loading.

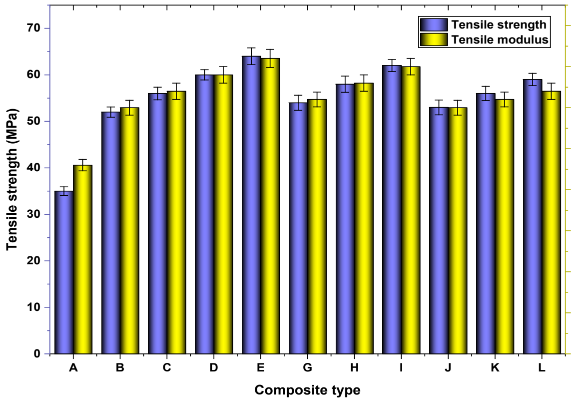

Figure 3. Tensile strength and modulus of hybrid hemp-based nanocomposites.

Overall, among the three nanofillers, it was observed that Al_2O_3 at 1.5 wt% loading marked the highest relative value of tensile performance and, therefore, served as the most efficient nanofiller in this investigation. This is in agreement with the literature, where it is cited that metal oxide nanoparticles like Al_2O_3 serve as preeminent fillers in polymer composites because of their high stiffness and capability to arrest cracks [19,20].

3.2 Flexural properties

The flexural properties of the prepared composite specimens were evaluated per the three-point bending test ASTM D790. The measurements obtained suggest that with the addition of hemp fibers and nanofillers, both the flexural strength and flexural modulus increased in comparison to plain epoxy, which indicates enhancement.

The epoxy resin with no additives (Sample A) exhibited a flexural strength of 60 MPa and a flexural modulus of 2.7 GPa. When hemp fibers were incorporated without adding nanofillers (Sample B), the flexural strength surged to 78 MPa and the modulus to 3.5 GPa. It can be attributed to the result of the fibers carrying bending stresses and restrictive deformation alongside the increase in load-bearing capability, leading to enhanced rigidity of the matrix. The addition of Al_2O_3 nanoparticles brought remarkable improvements to the flexural response. Sample C (0.5 wt% Al_2O_3) exhibited a flexural strength of 82 MPa and a modulus of 3.7 GPa, which further increased to 93 MPa and 4.2 GPa for Sample E with 1.5 wt% of Al_2O_3. This can be attributed to the rigid ceramic nature of Al_2O_3, which strengthens the matrix and provides good resistance to crack formation and propagation under flexural stress. Furthermore, the increased flexural modulus is associated with improved material stiffness, thus capable of resisting deformation under bending [21]. Figure 4 shows the flexural strength and modulus of hybrid composites.

The incorporation of SiO_2 nanoparticles (Samples G-I) also led to observed improvements in flexural properties, though lower than the Al_2O_3-based composites. With 0.5 wt% loading (Sample G), the flexural strength and modulus achieved were 80 MPa and 3.6 GPa, respectively. These values increased to 90 MPa and 4.1 GPa at 1.5 wt% (Sample I). The enhanced dispersion and surface area of SiO_2 enable better bonding with polymer chains, thus increasing the stress transfer at the fiber-matrix interface. While it is known that SiO_2 has lesser intrinsic hardness than Al_2O_3, the chemical compatibility along with the interfacial adhesion mechanisms helps suppress microvoids and reduce crack propagation, contributing to improved flexural characteristics. Among the additional fillers for the hemp/epoxy composite, MgO composites (samples J – L) had the least improvement in flexural properties. However, they still performed better than the neat hemp/epoxy composite. Sample J (0.5 wt% MgO) had a flexural strength of 77 MPa and modulus of 3.4 GPa, while sample L (1.5 wt% MgO) had 85 MPa and 3.8 GPa, respectively. About MgO nanoparticles, these mostly provide micro-filler effects and limited crack barrier capabilities while contributing to flexural reinforcement. Their moderate enhancements were likely due to the relatively lower stiffness and lesser surface reactivity compared to Al_2O_3 and SiO_2 [22]. In general, Al_2O_3-based composites led other types in both flexural strength and stiffness, while SiO_2-based systems followed closely behind. MgO-based composites showed fair improvement but were less effective at resisting bending deformation. This illustrates the dominant role that the type, size, and distribution of nanofillers have in the flexural behavior of natural fiber composites with regard to stress transfer efficiency, filler-matrix interaction, and crack resistance mechanisms. Fiber type also plays a vital role in improving the mechanical strength of hybrid composites. The reinforcement employed in this research was a 300 GSM plain-woven hemp fabric: a balanced plain weave structure of interlaced hemp, where the warp and weft yarns alternately cross and interlace at right angles. The uniform distribution of yarns in the fabric aids in achieving high dimensional stability and interfacial bonding with the matrix, resulting in uniform mechanical properties of the composite [23]. The balanced plain weave, in which warp and weft yarns interlace in a regular alternation, provides a high number of interlacing points. Although plain-weave fabrics typically exhibit higher yarn crimp, their balanced structure enhances load distribution and inter-fiber stress transfer, contributing to improved tensile and flexural performance. For instance, Sahbaz Karaduman *et al.* [24] reported that the plain weave (quasi-UD) performed best in tensile and flexural strength (second best overall) due to its balanced structure and efficient inter-fiber stress transfer compared to other weaves such as twill, basket, and quasi-UD.

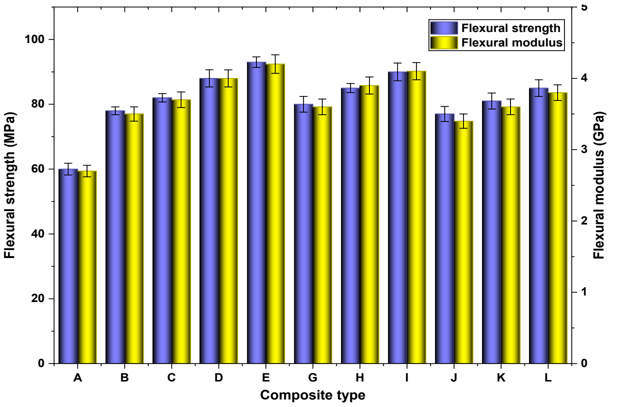

Figure 4. Flexural strength and modulus of hybrid hemp-based nanocomposites.

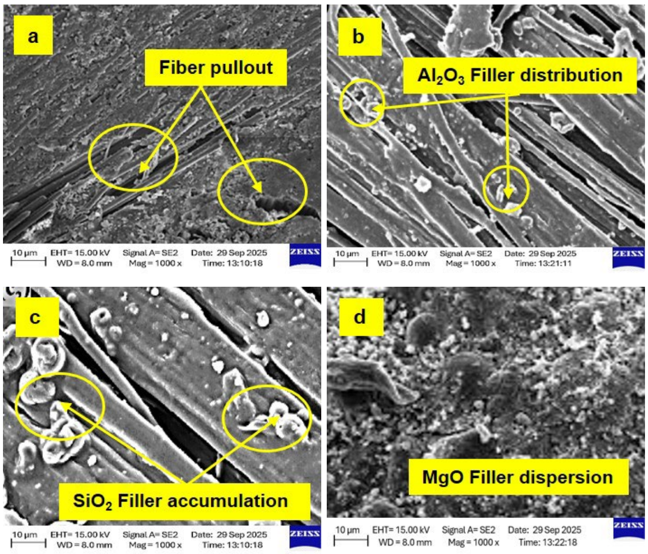

Figure 5. Microstructural analysis of (a) Neat hemp/epoxy composite, (b) Al_2O_3-filled composite (1.5 wt%), (c) SiO_2-filled composite (1.5 wt%), and (d) MgO-filled composite (1.5 wt%).

3.3 Microstructural analysis

The current research employed SEM micrography to analyze the microstructural features of the hemp fiber-reinforced epoxy composites in order to determine the effect of the nanoparticles Al_2O_3, SiO_2, and MgO on fiber-matrix adhesion, dispersion of the filler, as well as fracture characteristics, which are related to the tensile properties. The neat hemp/epoxy composite, as displayed in Figure 5(a), had fiber pull-out and interfacial gaps, which explain the lower tensile strength.

The use of Al_2O_3 nanoparticles improved the continuity of the matrix and the bonding between the fibers and the matrix, as seen in Figure 5(b). There is a uniform dispersion of the nanoparticles and visible crack-bridging, which correlates with the best tensile performance. The superior performance of Al_2O_3 is attributed to the high intrinsic stiffness and hardness of the material, along with excellent compatibility with the epoxy matrix and efficient stress transfer at the fiber–matrix interphase [22,23]. Also, its nanoscale size makes it more efficient in filling microvoids and impeding crack propagation, unlike SiO_2 and MgO. Figure 5(c) shows the SiO_2-filled composite, where relatively good dispersion of the nanoparticles is seen, reducing microvoids and improving interfacial contact, thus resulting in a moderate improvement in tensile properties. In the MgO-filled composite (Figure 5(d)), there was partial dispersion of the nanoparticles with slight agglomeration in some areas, which led to a less pronounced strengthening effect in comparison to Al_2O_3 and SiO_2. In general, the microstructural observations conform to the tensile test results and verify that well-dispersed nanoparticles, particularly Al_2O_3, improve load transfer and decrease crack propagation, thereby enhancing the mechanical properties of the hemp/epoxy composites [26]. At the microscale, these nanoparticles act as localized stress concentrators, promoting more uniform stress distribution throughout the composite. This behavior enhances load transfer efficiency, contributing to the observed improvements in both tensile and flexural performance [27].

4. Conclusion

The mechanical performance of hemp fiber-reinforced epoxy composites is remarkably improved by the addition of nanofillers. The unfilled hemp/epoxy system was outperformed by the Al_2O_3 nanoparticle composites on tensile strength by 23% and on flexural strength by 20% at 1.5 wt%. SiO_2 also filled the composites, which were at par on mechanical enhancement with the former achieving 62 MPa tensile and 90 MPa flexural strength at 1.5 wt%. The MgO composites showed modest enhancements with 59 MPa tensile strength and 85 MPa flexural strength at similar loadings. The SEM results provided evidence that the nano-fillers added assisted in the improved fiber–matrix adhesion with the reduction of micro-voids along with the prevention of crack propagation in the Al_2O_3-filled composites. When considering the fracture surfaces, the raised roughness and lowered fiber pull-out confirmed the greater burden-shedding effectiveness and ductility. These nano-reinforced hemp composites are suggested to have tailored mechanical properties coupled with strategic microstructural features, making them ideal for use in sports equipment, lightweight structural components, automotive interior panels, furniture, or green building materials. They serve as a competitive substitute to synthetic fiber composites due to their superior strength-to-weight ratio and biodegradability, claiming their merit in environmentally conscious engineering.

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