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The influence of deep eutectic solvent pretreatment on pineapple peel delignification

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ABSTRACT

Pineapple peel is recognized as a widely available lignocellulosic biomass that offers considerable potential for upgrading into valuable products. The removal of lignin, or delignification, is vital to improving cellulose accessibility for further biomass conversion. Recently, deep eutectic solvents (DES) have demonstrated effectiveness as green delignification agents. This environmentally sustainable method aligns well with contemporary initiatives to manage agricultural waste more responsibly while encouraging the adoption of cleaner processing protocols. This study investigates the delignification capability of DES on pineapple peel and performs structural characterization of the altered biomass. The study varied the hydrogen bond acceptor (HBA) and hydrogen bond donor (HBD) types and their molar ratios, using choline chloride (ChCl) and betaine as HBAs, and lactic acid (LA) and glycerol as HBDs. The molar ratios tested ranged from 1:2 to 1:9. According to Chesson-Datta analysis, the application of ChCl:LA at a molar ratio of 1:9 for one hour effected a 95% reduction in lignin and increased cellulose content to 65.54%. FTIR analysis confirmed the disappearance of lignin-specific peaks, SEM observations showed a more porous fiber morphology, and XRD measurements indicated a significant rise in crystallinity from 29.18% to 41.11%.

Keywords: Pineapple peel, cellulose, delignification, lignin, lignin removal.

INTRODUCTION

Pineapple peel biomass emerges as a highly abundant lignocellulosic residue from the global pineapple processing industry. Particularly in major producing nations such as Indonesia, the Philippines, and Brazil, annual pineapple production exceeds 28 million tons (Tran et al., 2023). This agricultural byproduct, comprising approximately 30–40% of the fruit's total weight, represents a substantial yet underutilized resource for biorefinery applications, including bioethanol production, nanocellulose extraction, and biochemical synthesis (Nasoha et al., 2023; Nath et al., 2023). The rigid nature of lignocellulosic biomass, arising from the intricate association of cellulose (35–50%), hemicellulose (20–30%), and lignin (20–30%), creates major obstacles to achieving efficient biomass conversion (Ashokkumar et al., 2022; Bytyqi et al., 2025). Lignin, a heterogeneous aromatic polymer, forms a protective matrix that shields carbohydrate polymers, thereby hindering enzymatic hydrolysis and chemical processing essential for value-added product generation. Effective pretreatment strategies are thus imperative to disrupt this matrix, selectively removing lignin while preserving cellulose integrity to enable downstream valorization pathways (Sharma et al., 2025).

Conventional delignification approaches, including alkaline and acid treatments, have demonstrated efficacy in lignin solubilization but are constrained by substantial environmental drawbacks. These methods generate hazardous effluents, demand high energy inputs for neutralization and wastewater treatment, and often cause indiscriminate degradation of holocellulose components,

compromising yield and quality of the resultant cellulosic material. Moreover, equipment corrosion and safety concerns associated with corrosive reagents limit their scalability for industrial biorefineries. In response to these limitations, DES has garnered attention as a sustainable alternative pretreatment medium. DES, formed through hydrogen bonding interactions between HBA and HBD, exhibits tunable physicochemical properties including low toxicity, biodegradability, and cost-effectiveness derived from bio-based precursors. These neoteric solvents facilitate lignin extraction by disrupting intra- and interchain hydrogen bonding interactions within the lignocellulosic matrix, promoting biomass swelling and enhanced solvent penetration without the volatility or flammability issues of ionic liquids.

This study systematically evaluates the delignification performance of tailored DES systems – comprising ChCl and betaine as HBAs paired with LA and glycerol as HBDs across molar ratios of 1:2 to 1:9 – applied to pineapple peel biomass. This study primarily seeks to assess lignin removal through Chesson–Datta analysis to select optimal DES, to characterize structural changes by FTIR, SEM, and XRD, and to evaluate pretreatment-induced modifications in the composition of cellulose, hemicellulose, and lignin. Through these analyses, the research establishes empirical correlations between DES molecular design and pretreatment outcomes, furnishing foundational data for scalable biorefinery integration.

MATERIALS AND METHODS

Materials

Primary feedstock was pineapple peel collected as waste in Prabumulih, Indonesia. The chemicals utilized included choline chloride, lactic acid, choline–glycerol, betaine, glycine, and distilled water, all of which were of analytical grade.

DES preparation

DES were prepared by mixing choline chloride with lactic acid, choline chloride with glycerol, betaine with lactic acid, betaine with glycerol, and glycine with lactic acid at molar ratios of 1:2, 1:3, 1:5, 1:7, and 1:9. The mixtures were heated at 80 °C under continuous stirring until clear, homogeneous liquids were obtained. The resulting DES solutions were then stored in tightly sealed containers at room temperature until use in the delignification process.

Delignification

Pineapple peel was cut into small pieces, dried in an oven at 105 °C for 1 h, ground, and then sieved to obtain a particle size of 60 mesh. A mass of 50 g of dried peel was placed into separate 1000 mL Erlenmeyer flasks. Deep eutectic solvent solutions were prepared in situ by adding hydrogen bond acceptors (choline chloride or betaine, 98%) to individual flasks and subsequently introducing hydrogen bond donors (lactic acid or glycerol, 98%) at various molar ratios. Each mixture was heated at 80 °C for 15 min under constant stirring at 500 rpm until a homogeneous liquid phase was obtained. The different DES formulations were added to the flasks until the pineapple peel particles were fully submerged, after which the flasks were placed in a shaking water bath to ensure thorough impregnation and homogenization of the slurry. The suspensions were then subjected to thermal treatment in an autoclave at 100 °C for 1 h. Following delignification, the slurry was washed with distilled water until the filtrate reached neutral pH, and the solid fraction was dried in an oven at 105 °C for 7–8 h.

Lignocellulose analysis

Sample characterization included the determination of cellulose, hemicellulose, and lignin contents using the Chesson–Datta method. For both the raw material and the DES-delignified samples, scanning electron microscopy was employed to observe surface morphology, Fourier-transform infrared spectroscopy was used to examine carbon–hydrogen bonding and lignocellulosic functional groups, and X-ray diffraction analysis was carried out to evaluate crystalline phases and to obtain information on unit cell dimensions, chemical composition, and selected physical properties of the samples.

RESULTS AND DISCUSSION

DES effects on lignin removal

Components' relative proportion in a DES is a critical parameter governing delignification

performance, as it directly influences the solvent's ability to disrupt lignin linkages within the biomass matrix (Song et al., 2024). Table 1. Shows the lignocellulose composition of untreated pineapple peel and treated samples with various DES. On the basis of the experimental data presented in Figure 1, the DES system composed of choline chloride and lactic acid at a molar ratio of 1:5 exhibited the highest delignification efficiency. This optimum ratio was identified from the combination of maximum lignin removal and the highest cellulose content achieved under the investigated conditions.

The choline chloride–lactic acid DES with a molar ratio of one to five was identified as the most effective formulation because it balances lignin solubilization with the preservation of cellulose fiber structure. The previous study found that, for cocoa bean shell, a ChCl:LA ratio of one to two provided an insufficient carboxylic acid concentration to form a robust hydrogen-bonding network with choline chloride, resulting in limited delignification (Benítez-Correa et al., 2023). Consistent with this, the study observed that a ChCl:LA ratio of one to three did not markedly enhance lignin removal from acacia dealbata wood (Magalhães et al., 2024), which they associated with the DES exhibiting a molecular weight similar to kraft lignin. In work on poplar wood pulp reported optimum delignification was reported at a ChCl:LA ratio of one to eight, giving a cellulose-enriched fraction of 68.28%, whereas further increasing the lactic acid proportion to a ratio of one to ten led to the formation of dark aggregates, attributed to excessive medium acidity (Li, Q et al., 2025). Such strongly acidic environments, caused by an overabundance of lactic acid, can promote lignin condensation and chromophore formation and simultaneously accelerate cellulose hydrolysis, thereby decreasing the cellulose content (Jiang et al., 2020). An excess of hydrogen bond donors can also perturb the internal equilibrium of the DES and favor the formation of aggregates that interact less effectively with lignin, reducing the overall solvating capacity (Bao et al., 2025). At the opposite extreme, when the hydrogen bond donor content is too low, the system cannot establish a sufficiently stable hydrogen-bonding network with choline chloride, which diminishes lignin dissolution efficiency (Benítez-Correa et al., 2023). Thus, using hydrogen bond donor ratios that are either excessively

Table 1. The composition of pineapple peel lignocellulose

DES	Ratio	Lignin (%)	Cellulose (%)	Hemicellulose (%)
Untreated	-	20.00	54.00	9.00
ChCl:LA	1:2	13.00	57.00	31.00
	1:3	16.00	34.00	30.00
	1:5	14.52	56.45	19.35
	1:7	15.56	45.56	20.00
	1:9	8.05	65.54	16.09
ChCl: G	1:2	7.50	62.00	10.50
	1:3	4.30	59.00	14.70
	1:5	3.86	59.00	33.00
	1:7	3.77	59.00	32.30
	1:9	1.90	60.00	22.10
Bet:LA	1:2	24.00	34.00	34.00
	1:3	17.00	38.88	35.00
	1:5	25.60	42.86	15.39
	1:7	14.90	60.70	9.00
	1:9	13.00	40.00	40.00
Bet:G	1:2	14.00	52.00	24.00
	1:3	18.00	54.00	23.00
	1:5	18.00	50.00	18.00
	1:7	12.00	57.00	5.00
	1:9	15.00	49.00	31.00

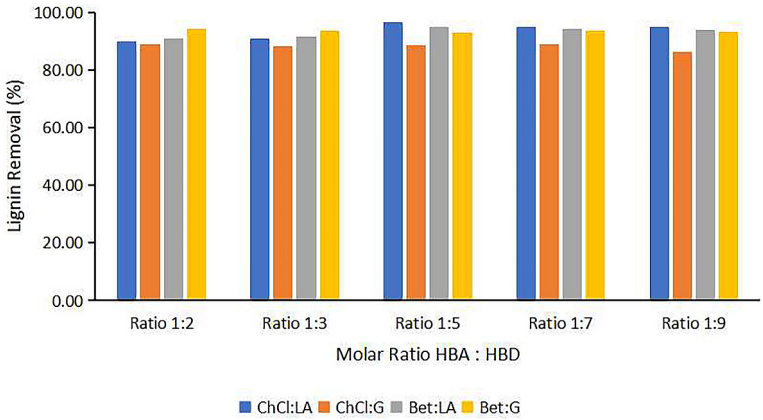

Figure 1. The effect of DES on pineapple peel lignin removal

high or too low can destabilize the DES system, promote unwanted degradation of cellulose and hemicellulose, and compromise both the selectivity and efficiency of the delignification process (Abolore et al., 2025).

SEM analysis

SEM was employed to observe and describe the surface morphology of pineapple peel before and after delignification. Figure 2 illustrates the morphological differences between the two samples. In Figure 2(a), the fiber surface is covered by a complex matrix layer composed of lignin, which acts as an inter-fiber binding phase (Baraka et al., 2025). This condition leads to occluded pores on the fiber surface, thereby limiting the accessibility of the underlying cellulose (Bai et al., 2022).

Figure 2(b) presents the microstructure of the sample following delignification with ChCl:LA DES at a 1:9 molar ratio, revealing pronounced alterations in the pineapple peel fiber structure. The fibers exhibit a more open surface, characterized by the appearance of pores and detachment of the binding layer, which leads to exposure of cellulose fibers (Cañadas et al., 2024). The emergence of these pores indicates that a substantial portion of non-cellulosic components, particularly lignin and silica within the cell wall, has been removed from the pineapple (Owhe et al., 2021). The observed results are consistent with prior observations that ChCl:LA-based DES is able to cleave β–O–4 bonds in lignin through a protonation mechanism (Li, P. et al., 2023). Moreover, the DES system facilitates the dissolution of silica and hemicellulose, thereby increasing fiber porosity and surface area. Consequently, the modified morphology enhances the accessibility of enzymes and chemical reagents in downstream steps such as enzymatic hydrolysis or cellulose nanocrystal production (Wang et al., 2022).

The increase in porosity and the opening of cellulose fibers in pineapple peel also enhance its capacity to absorb water and chemicals for use in subsequent post-delignification processes (Taib et al., 2025). The resulting higher hydrophilicity promotes fiber swelling, which directly enlarges the reactive surface area of cellulose (Kamdem Tamo et al., 2025). This condition is advantageous not only for downstream steps such as hydrolysis but also for other applications, including the fabrication of biopolymer-based composite materials. Increased porosity improves the matrix–reinforcement interface, i.e., the contact zone between the matrix phase (e.g., biopolymers) and the reinforcing phase (e.g., cellulose fibers). A better interface enhances adhesion between matrix and reinforcement, leads to more uniform stress distribution, and yields composites with higher strength and improved resistance to damage (Elfaleh et al., 2023). Thus, DES-based delignification not only alters the physical structure of the fibers but also improves the functional properties of pineapple peel for a range of industrial applications (Zheng et al., 2022).

Table 2 shows the composition of the elements of the sample treated and untreated. The decrease in C concentration (from 225.69 to 190.33) and O (from 83.57 to 77.31) following ChCl:LA 1:9 treatment indicates the removal of carbon-rich organic components, such as lignin, hemicellulose, and extractives from pineapple fibers, which is characteristic of delignification by deep eutectic

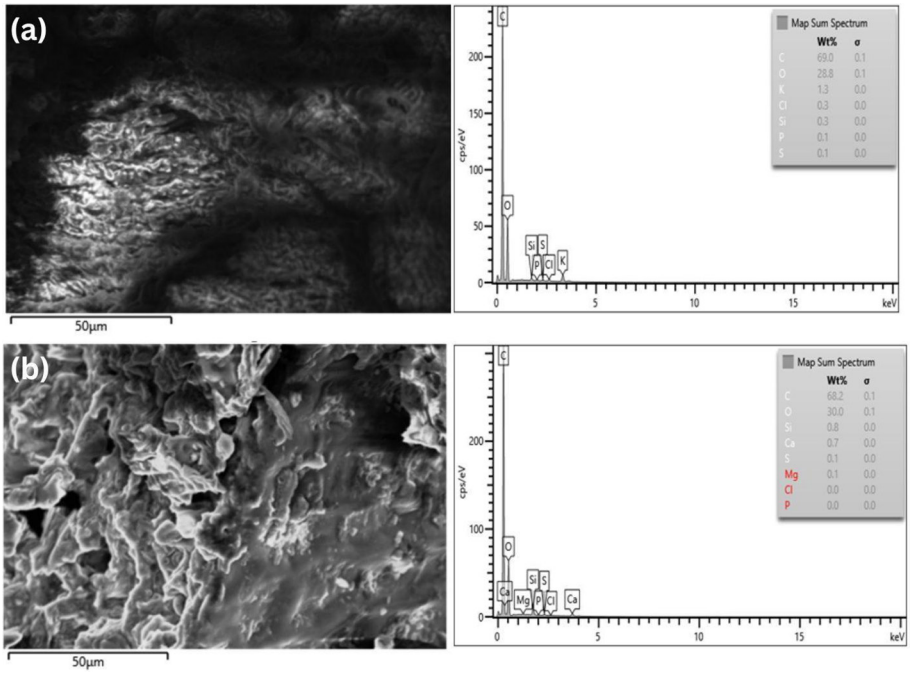

Figure 2. SEM of pineapple peel (a) untreated and (b) sample treated with ChCl:LA 1:9

solvents (DES). This observation aligns with established effects of DES on lignocellulosic biomass, where amorphous phases are preferentially solubilized. Similarly, the reduction in Cl (from 1.20 to 0.17) and P (from 0.96 to 0.24) reflects the dissolution of surface inorganic contaminants, including silica and soluble salts, in the lactic acid-based DES medium. The emergence of Mg (0.36%) and a substantial increase in Ca (2.73%) likely result from ion exposure during DES treatment, leading to mineral deposition or incomplete rinsing on the fiber surface post-processing. Sulfur levels remain stable (0.57–0.58%), suggesting resistance to DES extraction. These shifts are consistent with surface modification patterns observed in lignocellulosic treatments.

Table 2. The concentration of untreated and treated sample elements

Element	Untreated	Treated
C	225.69	190.33
O	83.57	77.31
Si	1.19	3.40
Mg	-	0.36
P	0.96	0.24
S	0.58	0.57
Cl	1.20	0.17
Ca	-	2.73

FTIR analysis

FTIR spectral analysis of pineapple peel revealed distinct differences in chemical characteristics between the untreated sample and the sample delignified with ChCl:LA (Figure 3). In the untreated sample, the O–H stretching band associated with cellulose hydroxyl groups appeared at 3400 cm^{-1}. After delignification, the increased absorption intensity at this wavenumber indicates greater exposure of cellulose O–H groups due to the reduction of lignin and hemicellulose fractions, which is further supported by the absorption band around 1040 cm^{-1} corresponding to C–O stretching vibrations of cellulose.

The absorption bands in the 1510–1600 cm^{-1} region show clear differences between samples before and after delignification. In the untreated sample, the FTIR spectrum exhibits a characteristic lignin band attributed to aromatic C=C vibrations. The reduction of this band after treatment indicates a decrease in lignin content within the biomass matrix. The agreement between FTIR data and quantitative analysis confirms that the DES system selectively degrades lignin within the lignocellulosic network. Changes in the intensity and position of absorption bands further demonstrate that delignification with ChCl:LA DES reduces hemicellulose and lignin while increasing the relative proportion of cellulose in pineapple

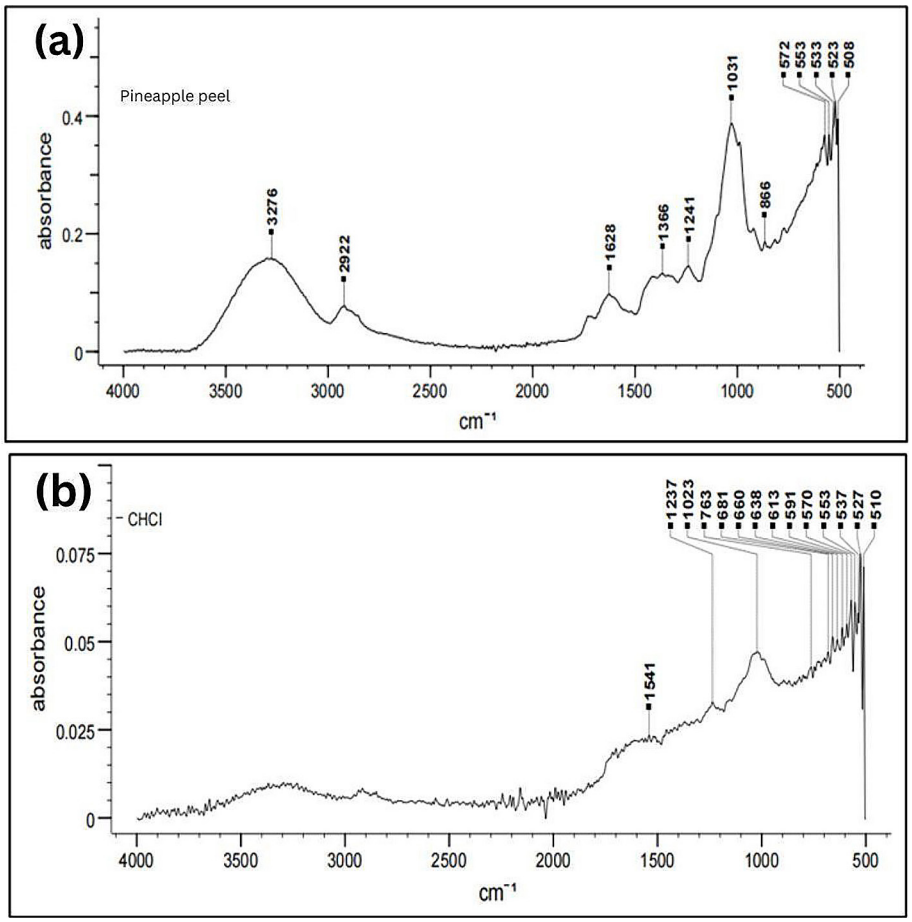

Figure 3. FTIR of pineapple peel (a) untreated and (b) sample treated with ChCl:LA 1:9

peel. The enhanced O–H, C–H, and C–O bands, together with the diminished C=O and aromatic C=C absorptions, provide evidence that cellulose becomes more concentrated following treatment.

XRD analysis

Figure 4 presents the XRD patterns of pineapple peel before and after delignification. The untreated sample shows diffraction peaks at 2θ = 34.96°, 44.64°, and 65.17°, with a crystallinity index of 29.18%. Following delignification, the peaks shift to 2θ = 44.68°, 49.25°, 72.69°, and 88.41°, accompanied by an increase in crystallinity to 41.11%. This enhancement is attributed to the removal of amorphous lignin, which makes the crystalline cellulose phase more dominant. Higher crystallinity is positively associated with fermentability, as purer cellulose is more readily hydrolyzed to glucose, and also indicates that pineapple peel has good potential as a feedstock for industrial applications requiring consistent fiber quality and adequate mechanical strength (Ahmed et al., 2022).

The observed increase in crystallinity also indicates that DES-based delignification not only dissolves non-cellulosic components but also preserves the integrity of the crystalline cellulose structure (Othman et al., 2025). In contrast, treatment with strong alkaline solvents such as NaOH can lead to cellulose chain degradation and a consequent reduction in crystallinity (AL-Rajabi et al., 2024). DES can therefore be regarded as a more selective medium, capable of disrupting lignin–hemicellulose linkages without damaging cellulose. This has a positive impact on the mechanical performance of the fibers, since a higher degree of crystallinity is generally correlated with improved tensile modulus and stiffness in cellulose-based materials (Jorda et al., 2022). High crystallinity is also crucial for advanced applications such as the production of cellulose nanocrystals (CNCs) or cellulose nanofibers (CNFs), where feedstocks with crystallinity values above 90% yield CNCs with a purer crystalline fraction and enhanced optical, thermal, and mechanical properties (Serrano-Martínez et al., 2025).

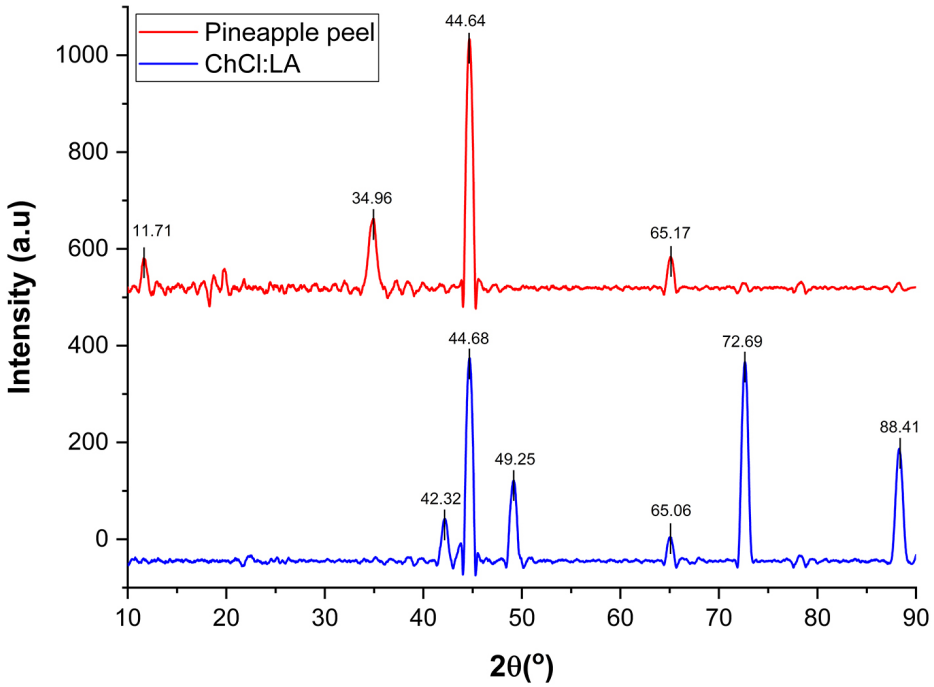

Figure 4. XRD of pineapple peel (a) untreated and (b) sample treated with ChCl:LA 1:9

CONCLUSION

ChCl:LA-based DES has been demonstrated to function as an efficient and environmentally friendly solvent for delignifying pineapple peel. The process achieved lignin removal of up to 95% and increased cellulose crystallinity from 29.18% to 41.11%. Evidence from FTIR, SEM, and XRD analyses indicates that lignin was removed while preserving the cellulose framework. Consequently, this pretreatment approach holds considerable promise for sustainable bioethanol production from agro-industrial waste.

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