

EFFECT OF INORGANIC ALTERNATIVE SCOURING AGENTS ON STRUCTURE OF CELLULOSE/POLYESTER BLEND FABRIC

A. U. Barambu^{1*}, D. E. A. Boryo¹, H. M Adamu¹, K. M yahuza¹, A. Abdulkadir²

¹Department of Chemistry, Abubakar Tafawa Balewa University Bauchi, Bauchi State, Nigeria

²Department of Chemistry, Gombe State University, Gombe State, Nigeria

*Corresponding author e-mail: aubnabiu@gmail.com, +2348032985884

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Abstract

In this research, a structural modification for a cellulose/polyester blend has been carried out using NH_4OH , $(\text{NH}_4)_2\text{C}_2\text{O}_4$ and liquid NH_3 which are environmentally friendly alternative scouring agents with NaOH as control. The scouring process was carried out on the samples with these alternative agents. Investigation into structural modification of cellulose/polyester blend fabric using NH_4OH , $(\text{NH}_4)_2\text{C}_2\text{O}_4$ and liquid NH_3 and its structural characterization with X-ray diffraction was carried. The results showed the inferences variation in the crystallinity index of scoured samples from (3.21-65.30%), the crystallite size of scoured samples (1.9-15 nm) in the crystalline region and (7-20 nm) in the amorphous region, inter-planar spacing of the scoured samples (0.340-0.350 nm) of the crystalline region and (0.350-0.340 nm) of the amorphous region and number of crystalline planes of the scoured samples is (3-11) of the cellulose/polyester blend fabric samples via X-ray diffraction studies. Among these alternative agents $(\text{NH}_4)_2\text{C}_2\text{O}_4$ and NH_4OH showed better interaction with both amorphous and crystalline regions of the cellulose/polyester blend fabric samples without loss in crystallinity when used as scouring agents, and the possibilities of being a superior alternative with significant effect on the structures of the cellulose/polyester blend, while the other samples showed possibilities of being highly competitive with the conventional agent.

Keywords; Cellulose, Inorganic, Polyester, Structure, Scouring Agents

Introduction

Cellulose today is the most used textile fibre in the world. Its current share is 56 percent for all fibres used for fashion and home furnishings in market [1]. Cellulose is a carbohydrate which is the chief component of the cell walls of plants and is found in wood and in cotton, linen, jute, hemp, leaves and stem fibers [2]. It is a basic raw material in the manufacture of rayon, acetate and triacetate fibers [3], and contains 44.4% carbon, 6.2% hydrogen and 49.4% oxygen. The elementary unit of a cellulose macromolecule is anhydro-d-glucose $\text{C}_6\text{H}_{10}\text{O}_5$, which is repeated severally in the cellulose molecules. Hence, it is a high molecular mass compound with d-glucose anhydrides of β -form are interconnected by 1-4 glycosidic linkage [4].

Polyester fibres are synthetic fibre with a long chain polymer composed of at least 85 % by weight of an ester of dihydric alcohol and terephthalic acid [5].

Dihydric alcohol like ethylene glycol react with an aromatic acid like terephthalic acid with two acid groups, the reaction can take place under suitable condition to make polymeric esters "polyester". One OH group of the alcohol molecule reacts with one COOH group of the terephthalic acid and the remaining COOH group of the acid reacts with another alcohol, thus react alternatively with alcohol and acid to form a chain of polymeric polyester [6, 4].

Cellulose/polyester blend fabric is the combination of natural and synthetic fabrics so that the good quality and properties are emphasis and a poor quality and properties are minimize. The blends of cellulose/polyester have developed into one of the most important textile fabric group. This is due to the unique properties of the fabric which has the potential to be similar in comfort performance to cellulose, but, owing to the presence of the polyester, be of a more stable price (possibly cheaper) than

cellulose and possess excellent easy-care properties [7]. Today, everyone needs better quality fabric products with improved performance at an affordable rate [8].

The modifications of these fabrics structure can be achieved by substituting the protons in the hydroxyl groups of cellulose to a varying extent using various substituents in production processes and chemical treatments [9, 10]. However, there was no detailed information on the structural changes (modification) in the fabrics. Hence, an investigation of this study is undertaken to know the actual changes that occurred on the structural property related. Also, to establish whether the modifications are on physical or chemical changes by the alternative agents on the fabrics during the scouring processes using X-ray diffraction.

Materials and Methods

Sample collection and preparation

The sample of cellulose/polyester blend fabric (3.5:6.5) was purchased from Central Market Bauchi, Bauchi State, Nigeria. The sample was cut into pieces of dimension 10 cm length by 10 cm width and kept in the laboratory prior to chemical treatment.

Scouring process (alternative scouring agents)

The pieces of fabrics samples were immersed into five different beaker containing equal volumes of 1 %, 2 %, 3 %, 4 % and 5 % NaOH solution after boiling for 5 minute. The samples were then allowed to boil for one hour using glass rod to completely immerse the fabrics into the solution. The fabrics were rinsed with cold over flowing tap water. 1 % acetic acid was used to neutralize the fabric followed by washing in 2 % detergent solution. Then the fabric was rinsed with distilled water and dried in the laboratory at room temperature. The same procedure was repeated for liquid ammonia, ammonium hydroxide and ammonium oxalate for the cellulose/polyester samples. 1% sodium hydroxide solution or 1% acetic acid was used to neutralize the different scouring agents [9].

Bleaching process (alternative scouring agents)

All the scoured samples were bleached using 4g/l sodium chlorite (NaClO_2) solution according to the procedure described by [11] and employed by [12, 13]. The pH of the sample was monitored using concentrated nitric acid (HNO_3) at a pH of 3 during preparation. The sodium chlorite solution was boiled on a hot plate for 5 minutes in a beaker, and the scoured fabric was immersed in the solution and allowed to boil for 3 hours at a temperature of 75°C after which it was washed in a hot water, and neutralized with 5% acetic acid, then washed with 2% detergent solution and finally rinsed with cold water and dried in the laboratory at room temperature for 45 minutes.

Mercerization process (alternative scouring agents)

In this research work mercerization process was carried out using concentrated solutions of 22% NaOH at a temperature 5°C for 45 minutes.

Preparation of samples

Fabric samples were conditioned in an oven at 80°C for 48 hours to remove moisture present at the surface and absorbed within the fabric samples. The samples were then blended using a manual blender.

XRD analysis of samples

Powder x-ray diffraction analysis was used to determine the crystalline and amorphous nature of the fabric samples. Powder XRD data were collected using an X-ray diffractometer (Bruker AXS D-8 Advance, Germany), equipped with Cu radiation source (wavelength $\lambda = 1.54056 \text{ \AA}$) operating at 40 kV and 40 mA and a Vantec detector.

The crystallinity index was estimated in this study using the equation adopted by [14] as shown below in Equation 1.

$$\text{CI} (\%) = \frac{I_{c+a}}{I_a} \times 100\% \quad (1)$$

Where; I_{c+a} is the intensity of the crystalline and amorphous peak, I_a is the intensity of the amorphous peak.

The crystallite size was determined using scherrer's formula which is as shown below;

$$\text{Crystal size (D) (nm)} = 0.89\lambda/\beta \cos \theta \quad (2)$$

The inter-planar spacing was determined using Bragg's formula [14] Equation (3);

$$n\lambda = 2d \sin \theta, \text{ where } n = 1. \quad (3)$$

$$\text{Interplanar spacing (d-spacing) (nm)} = n\lambda/2\sin \theta$$

Where; λ is the X-ray wavelength (0.154 nm), β is the angular width at half maximum intensity determine with the aid of Gaussian fit of the peaks on the diffractograms of the samples and θ is the Bragg angle.

Results and Discussion

Effect of inorganic alternative scouring agents on percentage crystallinity index of cellulose/polyester blend fabric: The primary purpose of scouring pretreatment in fabric production is to remove impurities which include fat, oil, wax and nitrogenous matters that may interfere with the dyeing, finishing operation and mechanical properties of the fabric. In this study, the morphological transformation in cellulose/polyester blend fabric during scouring was investigated using 1 to 4 % NaOH (Figure 1a), 1 to 4 % liquid NH_3 (Figure 1b), 2 to 5 % NH_4OH (Figure 1c) and 1 to 4 % $(\text{NH}_4)_2\text{C}_2\text{O}_4$ (Figure 1d). The 2θ in appendix 5 shows broadness between 10 to 20° which is an indication that the blend is amorphous, while the sharp peak between 10 and 20 in figure 20 under appendix III indicates crystallinity in the blend. The crystallinity index (41.89-65.30%) of the fabric increased on scouring with increase in NaOH concentration except with 2 % NaOH. Boryo established that, this could be an indication of improved mechanical strength of the fabric as the crystalline region had more contribution to mechanical strength than amorphous region [9]. The observed variation in crystallinity index could have resulted from either dissolution of the amorphous region or phase transition in the morphology of the scoured fabric, and it was confirmed.

Liquid NH_3 was able to interact with both the amorphous and the crystalline region as apparent from the crystallinity index (3.21-50.41%) of the fabric treated (figure 1b). The 2θ in figure 21 in appendix III shows broadness between 10 to 20° which is an indication that the blend is amorphous. The crystallinity index decreased to 0% when 3 wt.% liquid NH_3 was employed for the scouring process. Similarly, with 2 wt.% treatment, the crystallinity index was reduced but not to 0%. This observation suggested that liquid NH_3 interacted in a different manner when compared with that of NaOH. Unlike NaOH, liquid NH_3 was able to remove the impurities available in amorphous region more than crystalline regions of the cellulose/polyester blend. This brought about the observable reduction in the crystallinity index with increase in wt.% of NH_3 used in the scouring pretreatment. There was a significant variation in the crystallinity index from 1% to 5% (27.92 - 60.04%) (Figure 4c) of polyester fabric samples scoured with NH_4OH with the highest value observed at 4%.

In a similar version to liquid NH_3 , $(\text{NH}_4)_2\text{C}_2\text{O}_4$ was able to interact with both the amorphous and the crystalline region as shown from the crystallinity index (0.06 - 54.25%) (Figure 4d) of the polyester fabric blend samples treated. The crystallinity index decreased to 0.06% when 3 wt.% $(\text{NH}_4)_2\text{C}_2\text{O}_4$ was employed for the scouring process. However, with 4 wt.% pretreatment, the crystallinity index increased to 54.25% higher than the control while with 2 wt.% the crystallinity index was relatively constant with that of the control. This observations suggested that $(\text{NH}_4)_2\text{C}_2\text{O}_4$ interacted with the treated cellulose/polyester blend, fabric by penetrating both amorphous and crystalline regions like liquid NH_3 , as the penetration of the crystalline region brought about the observable reduction in the crystallinity index.

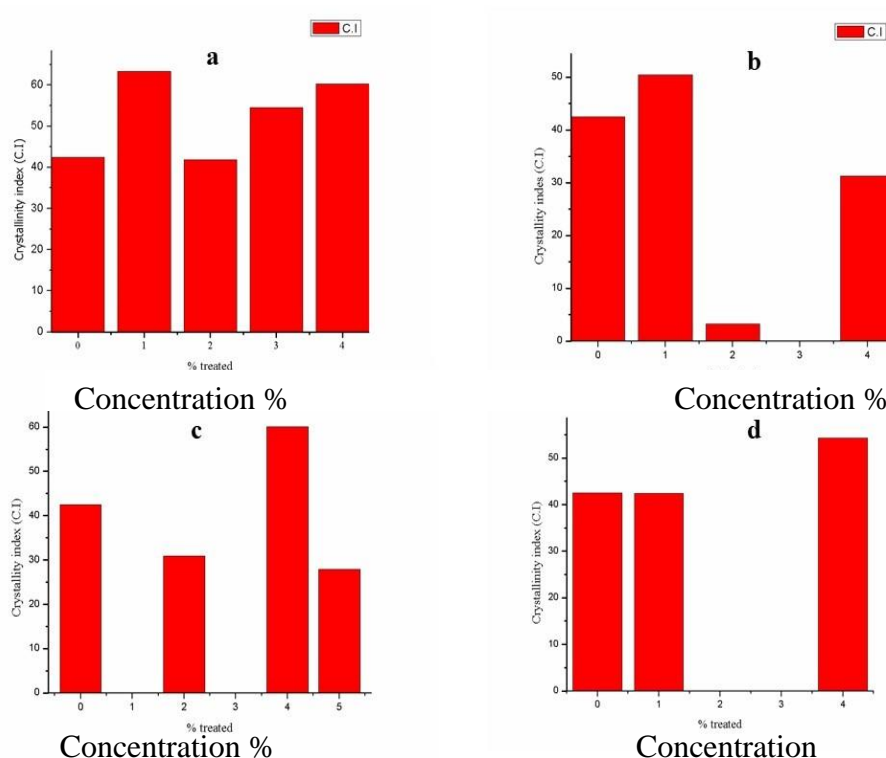


Fig. 1: Percentage crystallinity index for (a) NaOH, (b) Liquid NH₃, (c) NH₄OH, and (d) (NH₄)₂C₂O₄ scouring of cellulose/polyester blend fabric.

Effect of inorganic alternative scouring agents on d-spacing: The low concentrated NaOH (1 - 4 wt%) solution used in the scouring process contained hydrated species (NaOH.xH₂O) with large hydrodynamic radius, which could only penetrate the amorphous phase of the treated fabric. This could be easily noted in the difference in the inter-planar spacing (0.350 – 0.340 nm) of the amorphous and the crystalline region (Figure 2a).

The variation in the inter - planar spacing (Figure 2b) in the fabric scoured with liquid NH₃ was higher than that of fabric scoured with NaOH, an indication of improved interaction with the crystalline region by liquid NH₃ (0.348 - 0.340 nm), this could however lead to a compromise in the mechanical strength of the fabric. However, the interaction was more pronounced in the amorphous region as the inter-planar spacing (Figure 2c) of the crystalline region remained relatively constant (0.340nm), an indication of

higher resistance to penetration of the scouring agent. In comparison with NaOH and liquid NH₃, NH₄OH as a scouring agent was able to penetrate both amorphous and crystalline regions of the fabric although with less degree in the crystalline region. This showed better scouring properties without much influence on the morphology of the fabric thereby preserving the mechanical strength of the fabric. This observation aligned with findings of [9] on mechanical properties of alternative scouring agents where fabrics scoured with NH₄OH was reported to show improved mechanical property.

The variation in the inter-planar spacing (Figure 2d) in both crystalline region(0.344 – 0.350 nm) and amorphous regions was similar to liquid NH₃ however a steady progression was observed in the inter-planar spacing of the crystalline region compared to that of amorphous regio

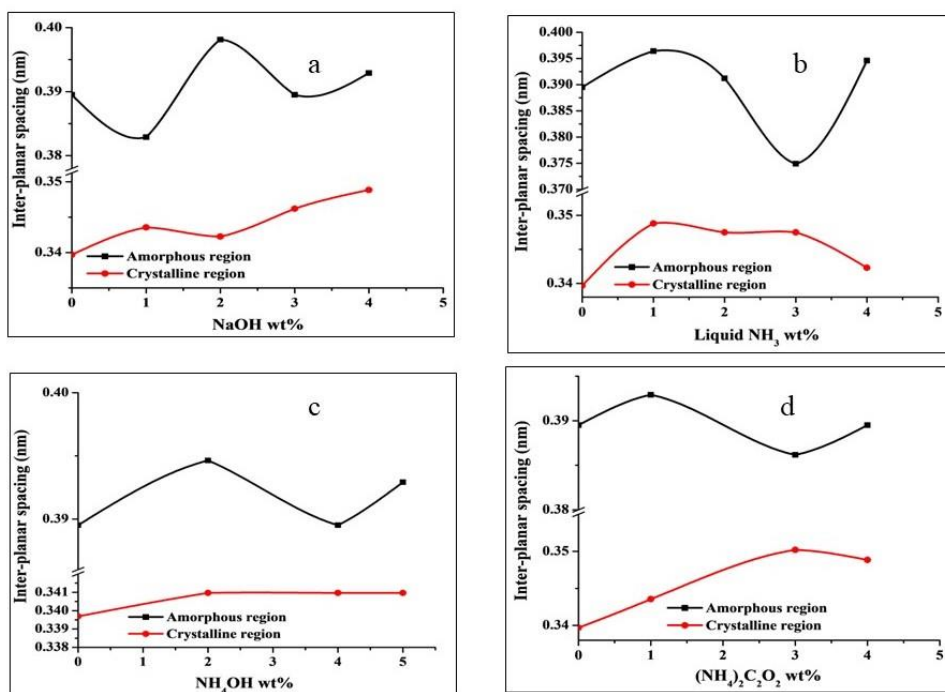


Fig. 2: The inter-planar spacing (d-spacing) for (a) NaOH, (b) Liquid NH₃ (c) NH₄OH and (d) (NH₄)₂C₂O₄ Scouring of Cellulose/Polyester Blend Fabric.

Effect of inorganic alternative scouring agents on crystallite size: The crystallite size (Figure 3a) of the amorphous region showed a reverse trend compared to that of the crystalline region (6.0 - 8.0nm) where the crystallite size increased and stabilized at high concentration of NaOH while the crystallite size of the amorphous region decreased with increase in wt.% of NaOH and from 2 wt.%, the crystallite size increased. This variation confirmed that conventional scouring process had stronger influence on the structural modification of the amorphous region compared to the crystalline region of the cellulose/polyester blend fabric. This agrees with the findings of [5, 9].

The observed downward trend in crystallite size (7.0 - 14.0 nm) (Figure 3b) of the amorphous region of the fabric scoured with increase in liquid NH₃ could suggest that some of the amorphous phase was affected just as the crystalline region was also affected in the reverse trend. This showed that liquid NH₃ affected both amorphous and crystalline regions of the cellulose/polyester blend fabric in a

competitive manner compared to NaOH that affect more with the amorphous region. This could have resulted in the improved dyeing properties of cellulose/polyester blend scoured with liquid NH₃ as reported by [5]

The trend observed in the crystallite sizes (1.9 - 8.0 nm) (Figure 3c) of both amorphous and crystalline regions in the fabric scoured with NH₄OH showed that the scouring agent interacted in a similar manner with the samples in both regions.

An inverse trend was observed in crystallite size (5.5 - 15.0 nm) (Figure 3d) of the amorphous and crystalline regions of the fabric scoured with increase in (NH₄)₂C₂O₄ suggesting that some of the amorphous region was affected just as the crystalline region was also affected in the reverse trend. This observation was similar to those observed in fabric scoured with liquid NH₃ and NaOH. This showed that scouring with (NH₄)₂C₂O₄ could lead to fabric with intermediate properties of those scoured with NH₃ and NaOH as observed by [9] in the optimum dry crease recovery.

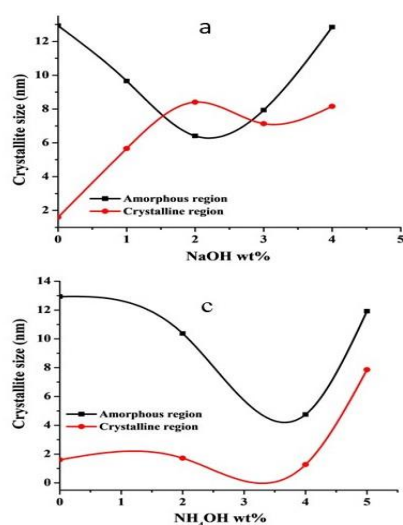


Fig. 3: The crystallite size for (a) NaOH, (b) Liquid NH_3 (c) NH_4OH and (d) $(\text{NH}_4)_2\text{C}_2\text{O}_4$ Scouring of Cellulose/Polyester Blend Fabrics

Effect of inorganic alternative scouring agents on number of crystalline planes: The effect of phase transition could also be observed in (Figure 4a) where notable changes were observed in the number of crystalline planes (6.0 - 11.0) with increase in concentration of the scouring

agent. As the concentration of NaOH increased, the number of crystalline planes increased and later reduced at 4 wt.% NaOH. This could be attributed to the presence of metastable phases in the morphology of the treated fabric.

Variation in number of crystalline planes (4.0 - 7.0) (Figure 4b) with increase in concentration of liquid NH_3 used for scouring also suggested presence of metastable state as observed in the fabric scoured with NaOH [11]. The crystalline phases were observed to be similar except at high concentration of 5 wt.% NH_4OH , the number of crystalline planes (3.0 - 7.0) (Figure 4c) was relatively constant. Similarly, steady progression was observed in the variation in number of crystalline planes (3.0 - 7.0) (Figure 4d) with increase in concentration of $(\text{NH}_4)_2\text{C}_2\text{O}_4$ used for scouring also suggested presence of metastable state as observed in the blend fabric scoured with liquid NH_3 and NaOH.

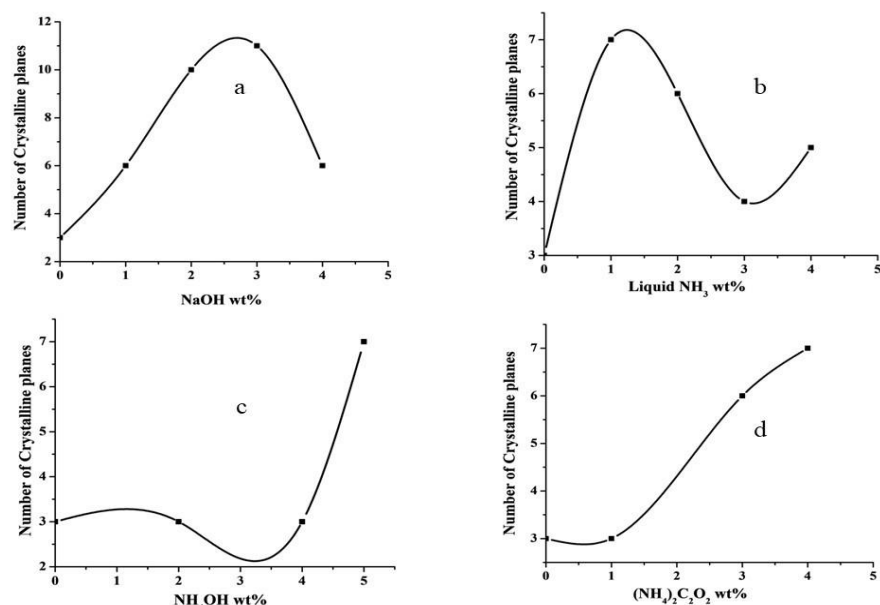


Fig. 4: The Number of Crystalline Planes for (a) NaOH, (b) Liquid NH₃ (c) NH₄OH and (d) (NH₄)₂C₂O₄ Scouring of Cellulose/Polyester Blend Fabric.

Optimum Conditions for Alternative inorganic Scouring Agents

Table 1: Optimum conditions for alternative scouring agents on percentage crystallinity index

Inorganic Scouring agent	Percentage crystallinity index(%CI)
2% NaOH	41.89
1% liq. NH ₃	50.41
4% NH ₄ OH	60.04
4% (NH ₄) ₂ C ₂ O ₄	54.25

Table 2: Optimum Conditions for Alternative Scouring Agents on d-spacing

Scouring agent	d-spacing (nm)
2% NaOH	0.340
4% liq. NH ₃	0.340

Table 1 all the optimum condition of the scoured agent for percentage crystallinity index are higher than that of the control (2% NaOH) Even though the d-spacing did not show much improvement compared to the control (0.340 or slightly higher, and 0.340 respectively) for the scouring agents as shown in Table 2. Table 3 and 4 all the optimum condition

4% NH ₄ OH	0.340
1% (NH ₄) ₂ C ₂ O ₄	0.344

Table 3: Optimum Conditions for Alternative Scouring Agents on Crystallite Size

Scouring agent	Crystallite size (nm)
2% NaOH	8.0
1% liq. NH ₃	14.0
4% NH ₄ OH	8.0
4% (NH ₄) ₂ C ₂ O ₄	15.0

Table 4: Optimum Conditions for Alternative Scouring Agents on Crystalline Plane

Scouring agent	Percentage crystalline plane
2% NaOH	10.0
3% liq. NH ₃	7.0
5% NH ₄ OH	7.0
4% (NH ₄) ₂ C ₂ O ₄	7.0

are higher than that of control with these all the alternative agent can computes with the control owing to it less hazardous effect and cheaper than NaOH.

Conclusion

The scouring of cellulose/polyester blend fabric using alternative agents was carried out and the effect on the structures of the

fabric were evaluated using X-ray diffraction. Among these alternative agents, $(\text{NH}_4)_2\text{C}_2\text{O}_4$ and NH_4OH showed the possibilities of being a superior alternative with significant effect on the structures of the cellulose/polyester blend. It was confirmed that the modifications on the fabric were both physical and chemical changes. Therefore, these alternative agents should be engaged as scouring agents industrially for commercial purpose.

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