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SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY OF PURE ZnO AND Cu DOPED ZnO NANOPARTICLES.

N.H.Bansod* and G.N.Chaudhari

Nanotechnology Research Laboratory, Department of Chemistry
Shri Shivaji Science College, Amravati - 444603 (MS). India.

Abstract:

The present study reports the synthesis of pure ZnO and Cu-doped ZnO (CZO) nanoparticles for structural and antibacterial applications. The samples were synthesized by simple, low cost, sol gel method. The characterization of synthesized nanocomposite were done by using various techniques such as X – ray diffraction (XRD) UV-Visible, Fourier Transform spectroscopy (FTIR), Scanning electron microscopy (SEM).

The antimicrobial activities of synthesized materials were determined against Gram-positive bacteria and Gram-negative bacteria by using disc diffusion method. The present study demonstrated that Copper doped ZnO (CZO) has remarkable antibacterial activity against all tested bacterial strains.

Corresponding Author:

N.H.Bansod,

Nanotechnology Research Laboratory,
Department of Chemistry
Shri Shivaji Science College,
Amravati-444603 (MS) India.
E-mail: nbansod15@gmail.com

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INTRODUCTION:

In recent times, the great deal of research has been conducted on ZnO nanomaterials with enormous scientific interest due to their vast attractive properties such as high isoelectric point (9.5), high band gap energy (3.37eV),**[1]** low cost, high stability, high value of binding energy of 60 meV.**[2]** A new methodologies has been developed, transition metal doped in nanomaterial having consistent effort for its excellent properties and versatile application. In the same context, a chief Transition metal doped in ZnO based nanomaterial leading to advancement in technologies and science such as, gas sensor, power generator, and field transistor.**[3-5]** Furthermore due to doping, the biocompatibility of ZnO has been enhanced.

Many studies have been reported the antimicrobial activity of ZnO nanoparticles against gram negative and gram positive bacteria such as Escherichia coli. *K. pneumoniae S. aureus E. faecalis.* **[6]** Moreover a low concentration of Cu dopant, a remarkable action against the bacterial strains.**[7-8]** Since ancient times the Copper nanoparticles has been attracted more attention as promising antibacterial agent.**[9]**

The aim of the present study is to enhance antibacterial behavior of ZnO nanoparticles by Cu doping. For this we have synthesized ZnO and Cu doped ZnO (CZO) nanoparticles by sol-gel citrate method and compared the structural and optical behavior as well as antibacterial activity of ZnO and Cu doped ZnO (CZO).

EXPERIMENTAL SECTION:

Preparation of nanomaterial

Synthesis of ZnO

Zinc oxide (ZnO) nanoparticle was synthesized by sol-gel citrate method.**[10].** Initially take Zinc nitrate [Zn $(NO_3)_2.6H_2O$] and Citric acid (C6H8O7), were dissolved in ethanol with 1:1:2 proportion. The mixture was stirred at 80°C for 3 hrs to get homogeneous solution was further heated at about 120°C for 12 hrs in pressure bomb to form the gel precursor. The prepared product was subjected to 3hrs heat treatment (calcinated) at 350°C in a muffle furnace. The dried powder was calcinated at 650°C in order to improve the crystallinity.

Synthesis of Cu doped ZnO (CZO)

Zinc nitrate, Copper nitrate (5, 10 & 15% by wt) and citric acid were dissolved in ethanol in stoichiometric proportion. The mixture was stirred on magnetic stirrer at 80^0c for 3 hrs till to get homogeneous solution and further heated in heating vessel for nearly 12 hrs at about 120^0c to get sticky gel. The prepared product was calcinated for 6 hrs at 650^0c in a muffle furnace. The overall scheme for synthesis of Cu doped ZnO (CZO) nanocomposite is shown in figure 1.

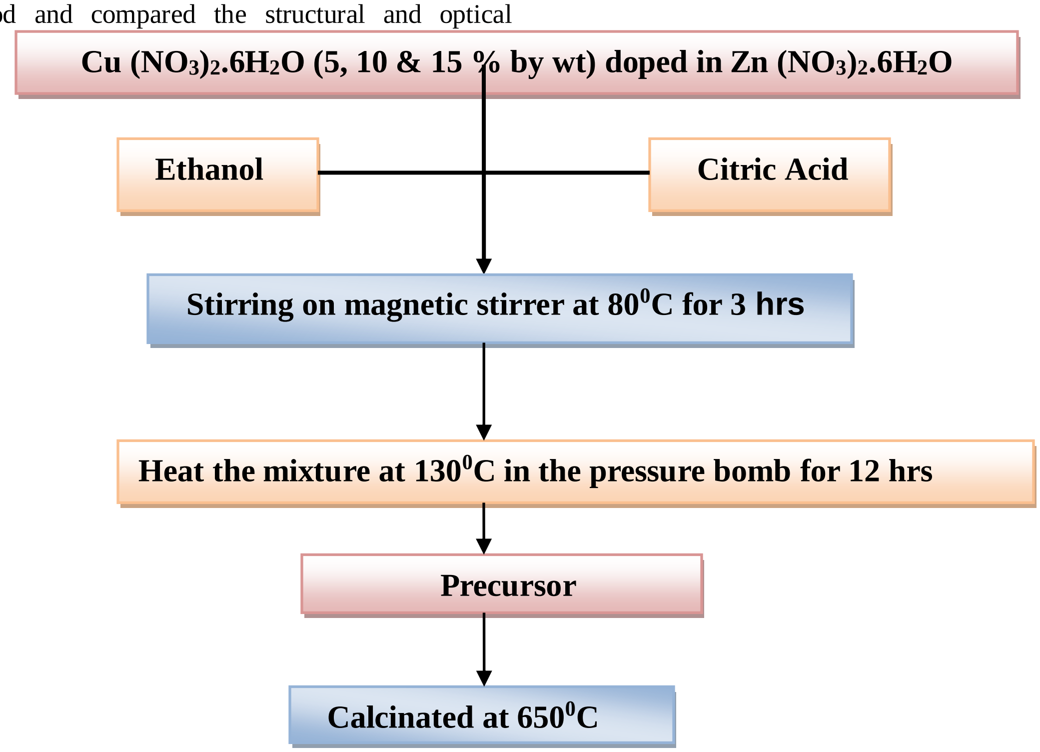

Fig. 1: Synthetic route for nanocomposite Cu doped ZnO

RESULT AND DISCUSSION:

X-ray Diffraction method

The powder x-ray diffraction methods are used to study the structural properties and the phase purity of the samples. **Fig.2.** shows the XRD spectra of the synthesized Cu-doped ZnO nanoparticles which is annealed at 650^0C. All the diffraction peaks have been labeled with (hkl) planes after comparing with JCPDS code (00-036-1451). The (101) peaks shows variation in the intensity at 36.29^0 Bragg's angle.

The Particle size of nanoparticle was determined using the Debye Scherrer equation.**[11]**

$$D = K\lambda / \beta \cos\theta$$

Where K is constant equal to 0.94, D and λ are the particle size in nanometers and wavelength of the radiation (1.54056 Å for Cu Kα radiation), respectively. β and θ are the peak width at half-maximum intensity (FWHM) and peak position.

Pos. [°2Th.]	FWHM [°2Th.]	Rel. Int. [%]
36.2929	0.3444	100

The particles size for CZO was found to be 47 nm which is lower than pure ZnO (48.4 nm). The decrease in the particle size was attributed to disorders created by the copper ions in the ZnO lattice structure. From the study it was assumed that for a smaller amount of Cu^{2+}, its ions substitute well with Zn^{2+} ions, but increasing Cu concentration causes a CuO cluster to form and isolate as an impurity phase.

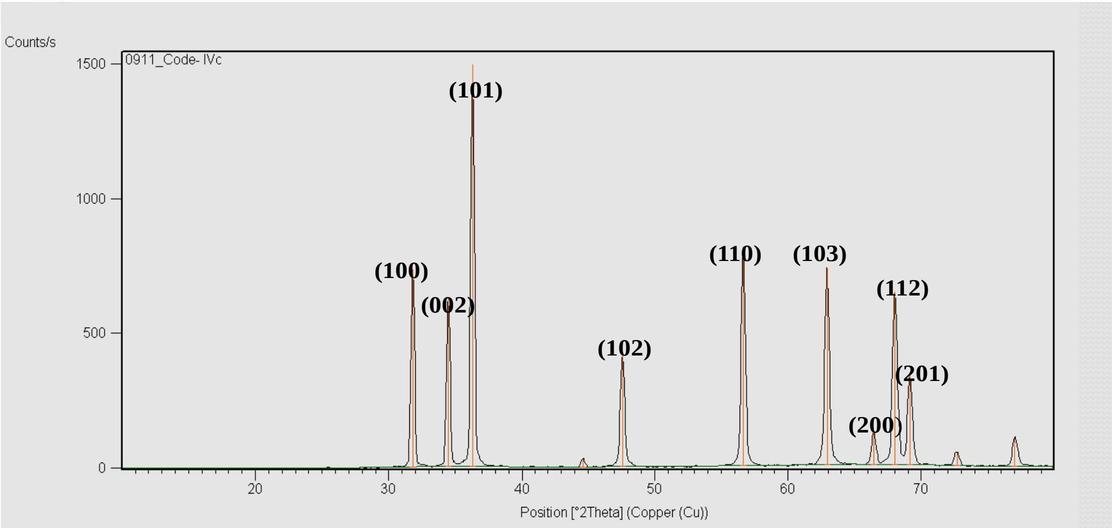

Fig.2: XRD pattern of 15% by wt Cu doped in ZnO

UV-Visible Spectroscopy

The UV spectra of Cu doped ZnO (CZO) are examined using (Shimadzu- 1800) UV-Visible Spectrophotometer in the visible range. **Fig.3.** represent the absorption spectra of Cu doped ZnO (CZO) at 378.0 nm in the visible range between 200 - 800 nm of wavelength, which indicate the formation of Cu doped ZnO nanoparticle by sol gel method.[6] The band gap energy is calculated on the basis of maximum absorption band of Cu doped ZnO. From the UV-Visible spectra, it was found that absorption band at 378.0 nm for Cu doped ZnO as compared to pure ZnO (374.7). The band gap energy is calculated according to following equation. **[12]**

$$E_{bg} = \frac{1240}{\lambda}$$

Where E_{bg} is the band gap energy and λmax is the wavelength (378.0 nm) of the nanomaterials. The energy band gap (E_{bg}) for Cu doped ZnO (CZO) was found to be 3.27 eV which is lesser than pure ZnO(3.30eV) Moreover, the absorption spectra is changed owing to incorporation of Cu^{2+} ions into the ZnO crystallographic planes.

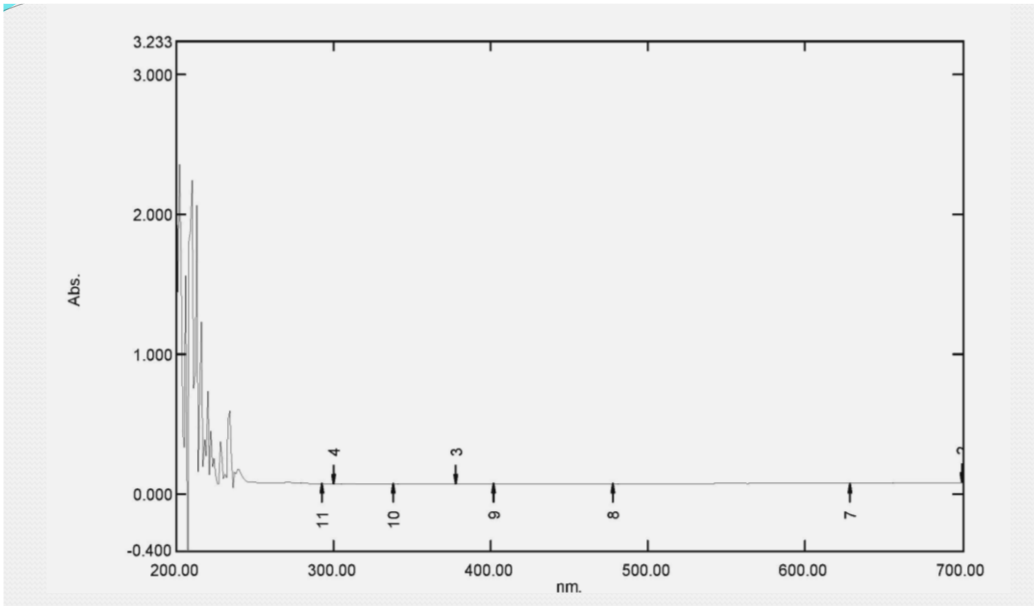

Fig.3: UV-Visible spectrum of Cu doped in ZnO

FTIR Spectroscopy

An FTIR spectrum was recorded using KBr solid pellet technique. **Fig .4.** Shows the FTIR spectra of Cu doped ZnO. The inset show some plot in the region 400-900 cm^{-1}. The weak absorption peaks at 3441 cm^{-1} and 1612 cm^{-1} are attributed to the O–H stretching vibrations and deformation of O-H bond respectively. Which are related to the absorbed water on the surface of nanomaterial. Another intense absorption peak at 432 cm^{-1} is related to the stretching vibrations of the Zn–O bond. Some absorption band near 613 and 652 cm^{-1} are also found. This is related to the vibration of the Cu–O bond.

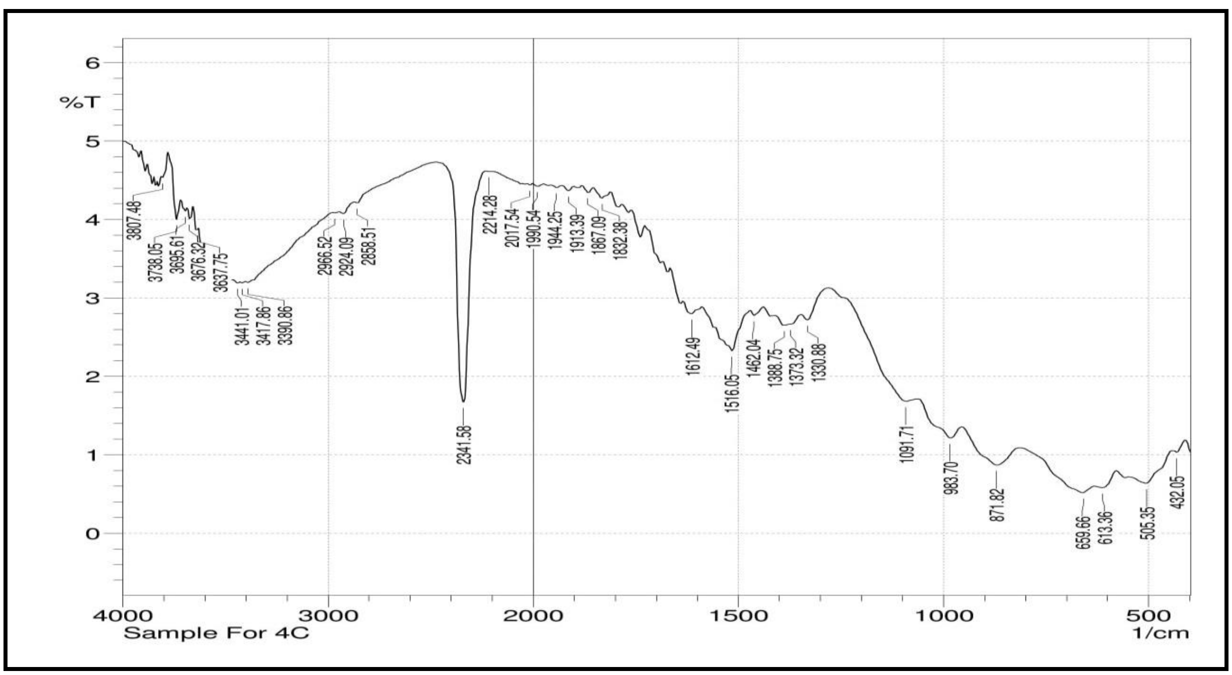

Fig.4: FTIR spectra of Cu doped in ZnO

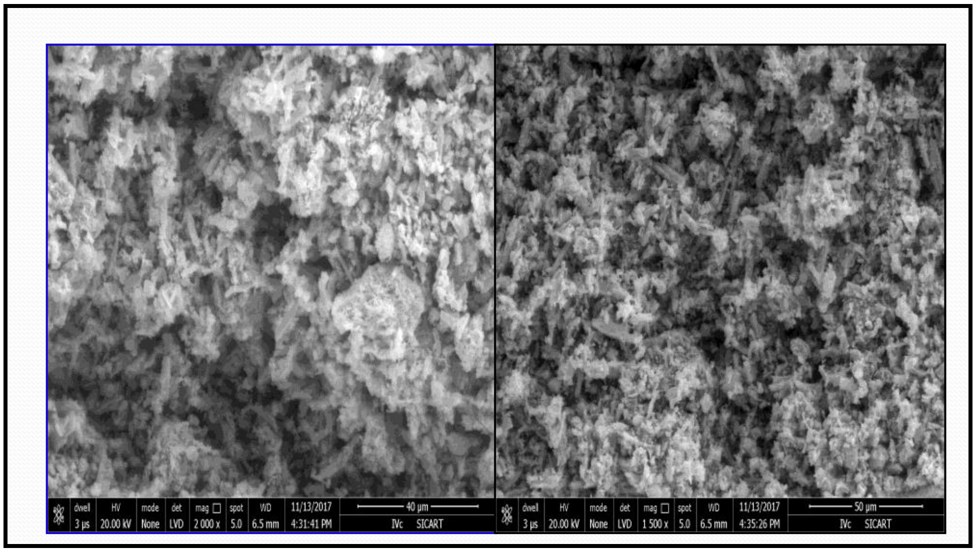

Fig.5. SEM spectra of CZO

Scanning electron Microscopy (SEM)

Fig.5. shows A typical micro –graph, has revealed the formation of homogeneous and uniformly dispersed nanoparticles. The Cu doping strongly influenced the grain size as well as morphology of ZnO nanomaterials. The average particles size has been decrease due to Cu doping in ZnO matrix.

Antimicrobial Activity

Disc diffusion assay

The nanoparticles pure ZnO and Cu doped ZnO (CZO) were screened for antimicrobial activity by using disc diffusion method. **[13]**. The bacterial organism were used included both gram positive and gram negative bacteria like *E. coli* (MTCC 118),*K. pneumoniae* (MTCC 109),*S. aureus* (MTCC 1430), *E. faecalis* (MTCC 2729). **Fig.6.(A,B)** reveals that Cu doped ZnO nanocomposite has remarkable antibacterial activity as compared to pure ZnO against test organisms.

Several mechanisms are responsible for the enhancement of antibacterial activity of Cu doped ZnO against bacteria, this may due to decomposition of ZnO and formation of oxygen reactive species. Another reason may be depicted as, due to decrease in size, nanoparticles are able to adhere on cell wall of organism, thus causing its destruction and killing. **[14-15]**

For experimental antibacterial susceptibility testing of nanoparticle, in the disc diffusion test, sterile Whatman filter paper (No. 1) disc were impregnated with 20µl of different samples,(1000 mg/ml) in DMSO. The disc were placed at the centre Mueller Hinton Agar seeded plates (M173) procured from Himedia Pvt. Ltd. Mumbai, with bacterial inoculums nearly 10^6 CFU/ml, incubated at 37^0C for 24 hrs. Then the growth free “zone of Inhibition” of respective disc was measured. The assay was performed in triplicate and mean value was considered as inhibition zone. Solvents were used as control and Show no inhibition in preliminary studies.

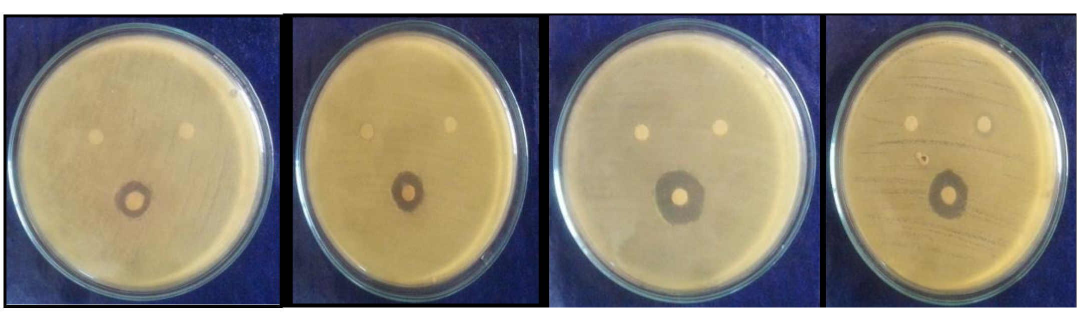

Fig.6. (A): zone of inhibition for CZO and pure ZnO against E.coli, S.aureas

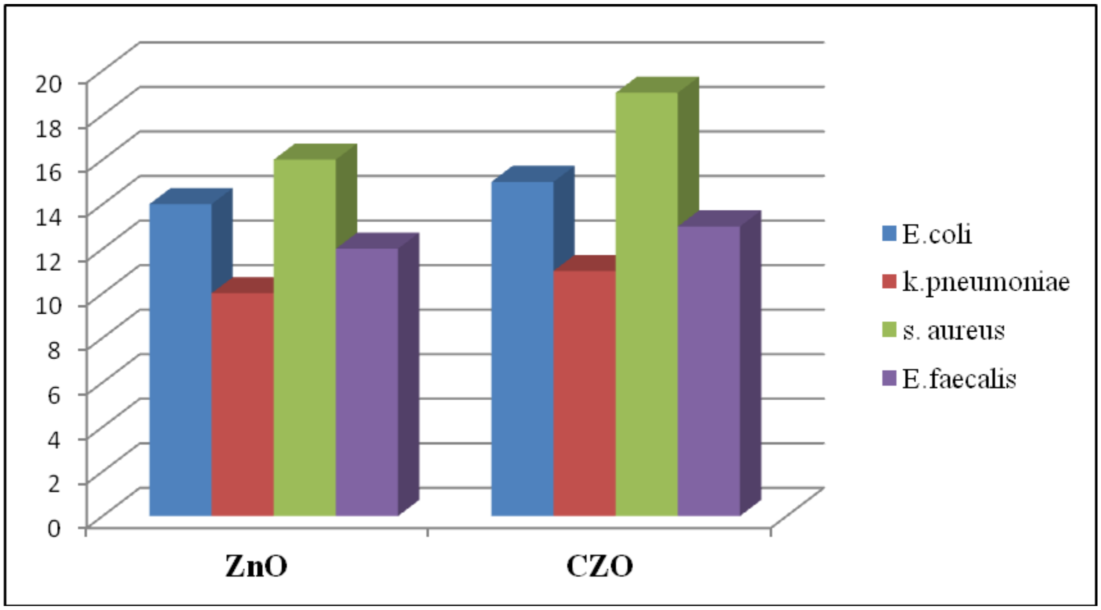

Fig.6. (B): Antibacterial activity of Pure ZnO and CZO

Determination of Minimum inhibitory concentration (MIC)

To evaluate minimum inhibitory concentration for CZO against all bacterial organisms. Take near about 40 µl of 0.2 mg/ml tri-phenyl tetrazolium chloride (TTC), an indicator was added in each bacterial suspension ((20 µl) and incubated for another 30 min. Inhibition of bacterial growth was confirmed visibly from a colourless well, while development of pink-red colour indicates the presence of bacterial growth. MIC values were recorded as the lowest concentration of sample at which bacterial growth is completely inhibited.

Sample	*E. coli* (MTCC 118)	*K. pneumoniae* (MTCC 109)	*S. aureus* (MTCC 1430)	*E. faecalis* (MTCC 2729)
CZO	90µg/ml	80µg/ml	60µg/ml	80µg/ml

From the values, it was found that Cu doped ZnO(CZO) nanocomposite has better MIC values than pure ZnO. Excellent MIC (60µg/ml) of 15% doped copper in ZnO (CZO) against S.aureus was obtained.

CONCLUSION:

In the present study, Cu doped ZnO (CZO) showed superior antibacterial activity against gram positive and gram negative bacteria than pure ZnO. The excellent MIC (60µg/ml) for CZO against S.aureus was obtained. The XRD results revealed that the decrease in particle size for CZO (47.0 nm) than pure ZnO, as well as from UV-Visible results, it cleared that band gap energy was decrease for CZO (3.27ev), this may attributed to Cu^{2+} ions replaced Zn^{2+} ion in the lattice. However Based on the finding of present studies, Cu doped ZnO(CZO) possessed enormous potential as an antibacterial agent and can be pursued important candidates for future studies.

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