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GREEN SYNTHESIS AND STUDY OF PHYSICAL PROPERTIES OF GOLD NANOPARTICLES

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ABSTRACT
The gold nanoparticles (Au- NPs) were prepared using the sumac aqueous extract as a reduction /stabilization agent by green synthesis as an eco-friendly method. Gold tetrachloride acid ($HAuCl_4.3H_2O$) was used in the synthesis of Au- NPs in (1 and 5) mM with the sumac aqueous extract and the mixture ratio was 1:1. Gold nanoparticles were characterized using X-ray diffraction (XRD), transmission electron microscopy (TEM), atomic force microscopy (AFM) and UV-Vis spectroscopy. From X-ray diffraction results demonstrate that Au- NPs were indexed into the cubic lattice with average crystalline size (67.36 and 69.42) nm at (1 and 5) mM respectively. TEM image showed that the shape of the particles spherical and cylindrical mixture with average size (25 and 77) nm for (1 and 5) mM respectively. AFM showed at concentration 1mM the average grain size distribution was 64.02 nm and for concentration 5mM was 69.29 nm.

KEYWORDS: AuNPS; Green synthesis; structural properties; optical properties and aqueous extract; eco-friendly.

INTRODUCTION

Nanotechnology is one of the quickest developing regions of science and innovation, the combination of metal nanoparticles is a functioning region of research in the field of nanotechnology with an exponential advance in biomedical applications including diagnostics, imaging, tranquilize conveyance and therapeutics utilizing metal nanoparticles[1,2]. A few chemical and physical techniques have been utilized for the preparation of nanoparticles, be that as it may, various strategies show disservice including the use of dangerous solvents, high-vitality use, unsafe items and so forth. Hence, there is a basic need to create condition agreeable strategies for a union of metal nanoparticles. The advancement of eco-friendly advances in the material blend is of extensive significance to grow their organic applications, these days, assortments of green nanoparticles with very much characterized substance arrangement, size, and morphology have been blended by various strategies and their applications in many imaginative mechanical regions have been investigated[3-5]. Because of their one of a kind optical, electrical and reactant properties, gold nanoparticles AuNPs have been utilized as a part of many everyday issues, predominantly in medication, hardware, and advances of assembling present-day materials[6-9]. AuNPs have been very used as specialists in biomedical identification and differentiating picture because of its properties that rematch: incompatibility, conjugation, and optical properties, it has small size, high surface region and soundness at high temperatures make them culminate device in medicinal diagnostics, hemodynamic treatment and in addition in the dynamic transport of medications, particularly for cancer treatment. AuNPs are for the most part unsteady as a result of their high surface and an appropriate stabilizer ought to be added to avert accumulation[10]. Sumac" red, red shift, turning red" is any of around 35 types of blooming plants in the class Rhus and related genera, in the family Anacardiaceous. The dried and powdered products of Rhus corsair are utilized as a flavor in Middle Eastern and South Asian food. Sumac develops in subtropical and calm locales all through the world, particularly in East Asia, Africa, and North America, see figure (1), the renewable nature of plant extricates, eco-friendly watery medium and mellow response conditions make the technique profitable over different perilous strategies. In the most recent years, diverse kind plants remove and their items have gotten consideration because of its minimal effort, vitality productive and nontoxic conduct in approach for the preparation of metal nanoparticles[11]. Many researchers have prepared gold nanoparticles using eco-friendly methods, where they studied the properties of AuNPs[12-18], and another section used nanoparticles prepared in environmentally friendly ways in the treatment of many diseases[19-23].

In this paper, we used a green synthesis method for the preparation of gold nanoparticles via the reduction of salt gold by aqueous sumac, without any extra additive protecting nanoparticles from aggregating. And study the structural and optical properties.

FIGURE 1: image of sumac's plant

MATERIALS AND METHODS

Etrachloroauric acid ($HAuCl_4.3H_2O$, with purity 99.98%) was purchased from Sigma Aldrich Chemicals. Freshly prepared distilled water was used during the experimental work and the sumac powder, where collected from the local market in Baghdad.

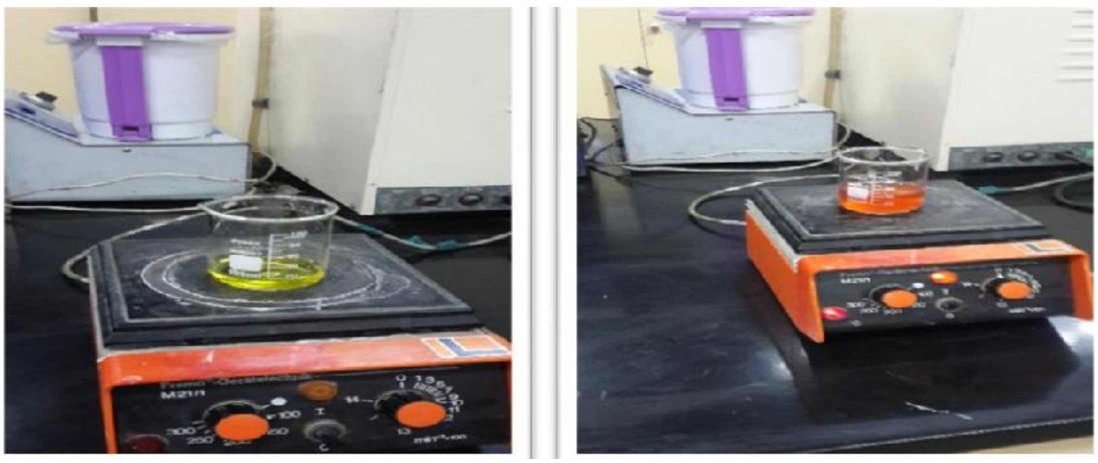


FIGURE 2: explains the change in color from pale yellow to orange

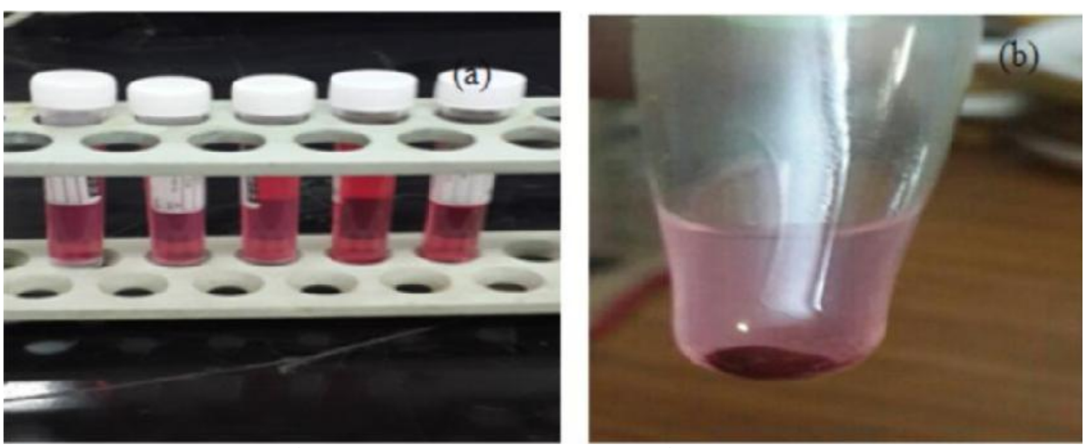


FIGURE 3: produced samples of AuNPs explain the change in color from orange to ruby red at (a) 5mM (b) 1mM.

Sumac aqueous extract was used as reducing agent, the aqueous extract was prepared by drenched 1gm of sumac powder in 100ml of distilled water using magnetic stirrer, and the mixture was boiled for 30 minutes at 200°C. The extract was centrifuged for 30 minutes at 4000 rpm; the aqueous solution was filtered by standard fresh filtration method. The extract was stored at 4°C for purpose use[24]. The gold nanoparticles were synthesized from 1 mM and 5 mM of $HAuCl_4.3H_2O$ by a magnetic stirrer. The mixture was heated for 60 minutes at 200°C and mixed with 20mL of sumac aqueous extract with stirring using a magnetic stirrer for 40 minutes at 100°C where the color change from pale yellow to orange as in figure 2, the product mixture was centrifuged at 4000 rpm for 30 minutes. Figure 3 shows the final product after leaving for one hour to change the color of the solution change to looks like a ruby red [25].

Instrumentation

Structural properties were analysis by using X-ray diffraction, the device used was Shimadzu-6000, made in Japan, the target is Cu, wavelengths λ=1.5406 °A, current 300 A, voltage 40 Kv, range of angle (10-80) degree, the UV-Visible spectroscopy was recorded at room temperature, the instrument using λ-Helios SP Pye-Unicam lamb with tube quartz, Transmission Electron Microscopy (TEM) analysis was made at a high-resolution electron microscope (HRTEM: JEOL JEM 2010) operating at an accelerating voltage of 200kV, AFM image was done to analyze the morphological surface of AuNPs, the sample prepared to test after drop-cost the solution of AuNPs on a glass base and dry by the used oven, the instrument type (Veeco Metrology, Autoprobe CP- II, Model No. AP0100) using silicon probes (RESPA-M, Veeco, Santa Barbara, CA.

RESULTS AND DISCUSSION

XRD analysis

X-ray diffraction image has been recorded from 35 to 70 as figures (4 and 5) which show the phase of gold nanoparticles at 1mM and 5mM respectively. The position of the peaks at 1mM were (111) and (220) which correspond to angles 38.21 and 66.19 respectively as shown in figure (4) which is refers to gold phase according to the comparison with standard cart (00-004-0784), from figure (5) shows the position of peaks at 5mM was (111) and (220) which correspond to angles 38.22 and 66.19. From figure (5) the predominant trend was toward (220) which are represented by the high intensity. The structure for two samples was cubic phase for Au NPs; the average crystallite size of gold was determined using Debye Sherrer formula [26].

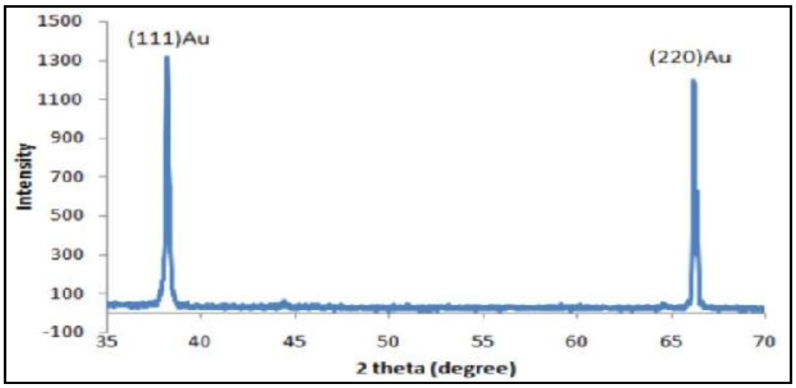


FIGURE 4: XRD pattern of AuNPs at 1mM.

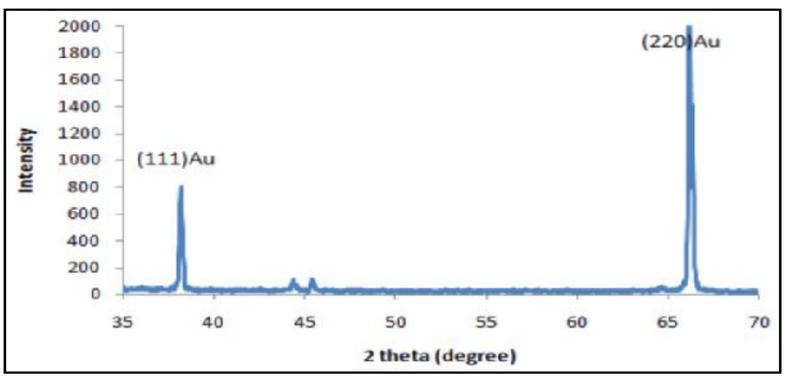


FIGURE 5: XRD pattern of AuNPs at 5 mM.

The average crystallite size (D) of AuNPs at 1 mM was 67.36 nm and the average crystallite size (D) of AuNPs at 5 mM was 69.42 nm. We can see from two figures the average crystallite size increased with the concentration of $HAuCl_4.3H_2O$ increase; this can be attributed to increased concentration, leading to increased aggregation. Table (1) summarizes the results of XRD parameters for (1 and 5) mM, these results correspond to a previous study (H. Eman, *et al*., 2014) [26].

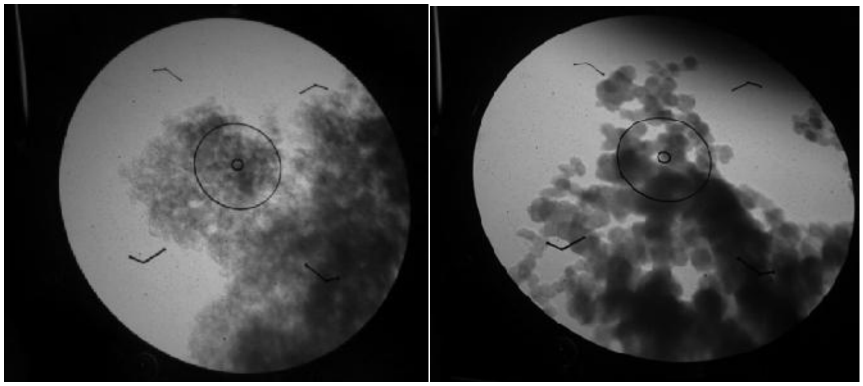
TABLE 1: XRD parameters of AU NPs for two concentration 1 mM and 5 mM

	2θ (degree)	FWHM (degree)	Crystallin size (D) (nm)	hkl
1	38.21	0.15	55.87	111
	66.19	0.12	78.85	220
5	38.22	0.14	60.00	111
	66.19	0.12	78.85	220

SURFACE MORPHOLOGY

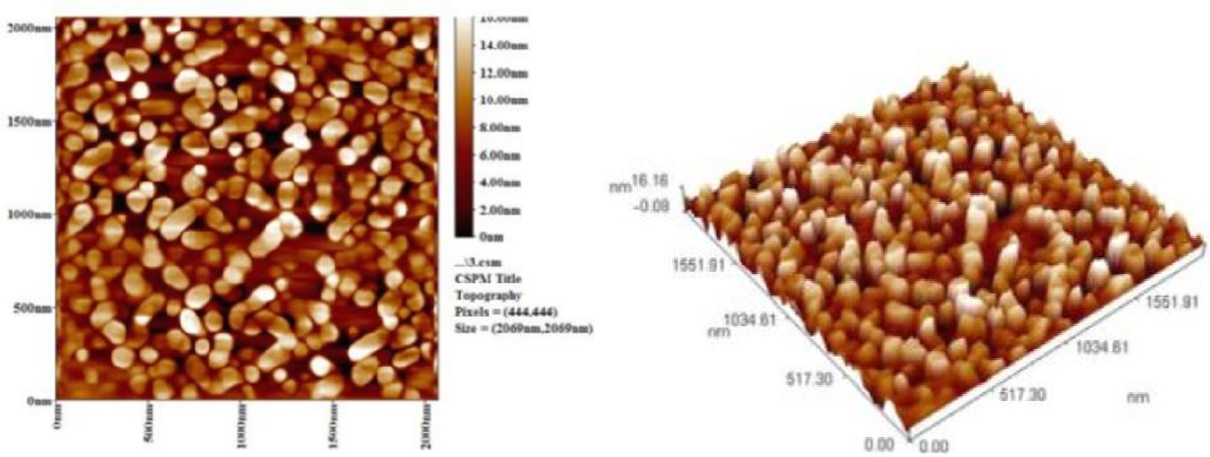
TEM analysis

Figure (6 a, b) shows TEM images of the gold nanoparticles prepared at 1 and 5 mM respectively, it can be seen that the grain forms are a mixture spherical and cylindrical . The TEM results show that the grain size for AuNPs at 1 mM was 25nm, while the grain size of the AuNPs at 5 mM was 77 nm, the results of the TEM analysis showed that the size of the particle increases with an increase in the concentration of $HAuCl_4.3H_2O$. These results correspond to X-ray diffraction results. Comparative analysis has shown that the relatively higher concentration ratio is responsible for fully symmetric nanoparticles [27, 28].

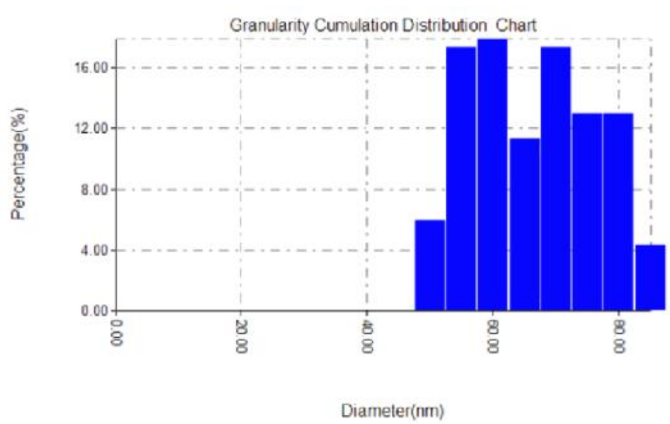


(a) (b)

FIGURE 6: TEM images of AuNPs at concentration (a) 1 mM (b) 5 mM



(a) **(b)**

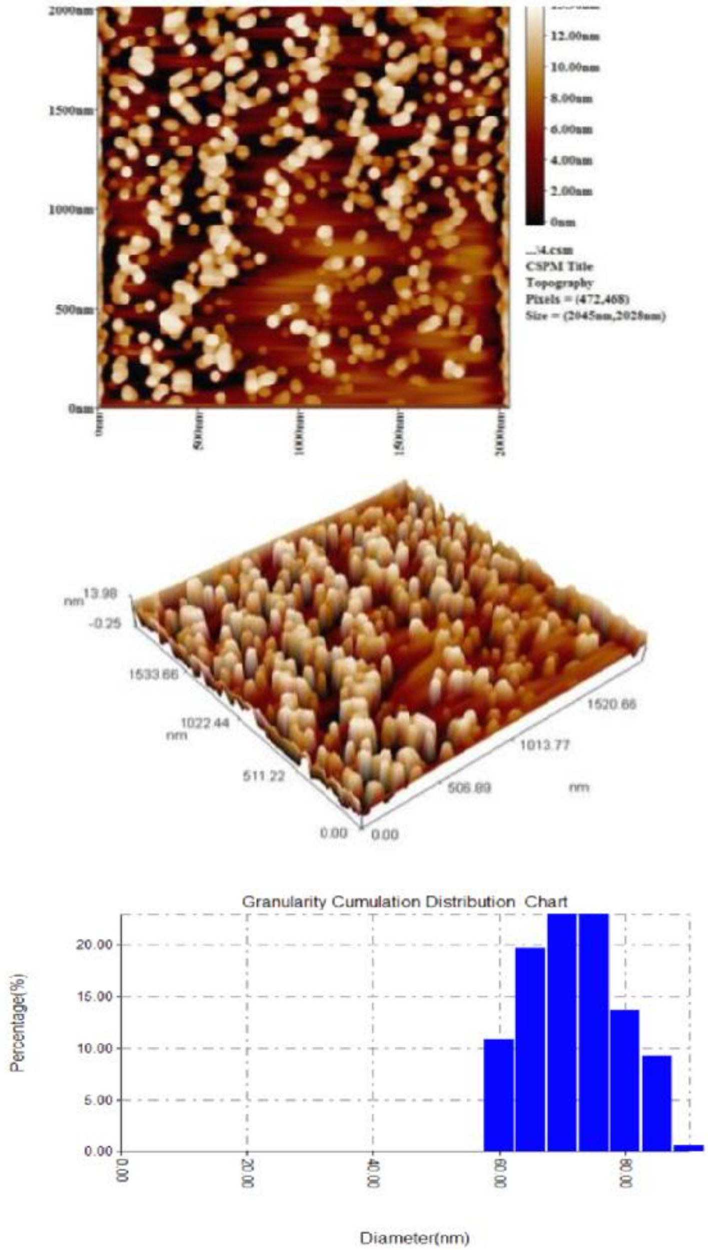


(c)

FIGURE 7: explain image of AFM of AuNps for concentration 1 mM, (a) two dimensions, (b) three dimensions, (c) histogram of distribution of grain size.

AFM images

Figures (7 a, b and c) and (8 a, b and c) shows images at two dimension, three dimension and a histogram distribution of grain size on the surface of prepared samples at 1 mM and 5 mM respectively ,the results of AFM measurements are listed in table (2), from the results we can see the value of vibration data for 5 mM higher than of 1 mM that is due to the increase in the concentration of $HAuCl_4.3H_2O$[26] ,these results correspond to X-ray diffraction and TEM results. Moreover, it can be seen from the figures (7 and 8) that the shape of the particles was a mixture between spherical and cylindrical this corresponds to the TEM measurements.



(a) (b)

(c)

FIGURE 8: explain image of AFM of AuNps for concentration 5mM (a) two dimensions, (b) three dimensions, (c) histogram of distribution of grain size.

TABLE 2: Average grain size, roughness and square mean root of gold nanoparticles

Concentration of $HAuCl_4.3H_2O$ (mM)	Average grain size (nm)	Roughness (nm)	Square mean root (nm)
1	64.02	3.19	3.86
5	69.29	3.53	4.07

UV-Visible analysis

Figure (9) shows the UV-visible spectra of produced samples of AuNPs, from this figure appearance of strong surface plasmon resonance (SPR) band absorption peak centered at about 503nm and 531 nm for 1mM and 5 mM respectively and show also that when the increasing of concentration from 1 to 5 mM leads to slightly shift towards higher wavelengths. From figure (9) it can be noticed that the width of SPR peaks for two concentrations was from 450 nm to 580 nm. This blue shift (130 nm) due to that particle is more spherical and size decreases[26]. Energy gap is determined by equation

$$E_g = h\upsilon/\lambda$$

Where E_g: energy gap(eV), hυ: photon energy (eV), λ: wavelength (nm).The results of energy gap showed the value of concentration 1 mM was 2.46 eV and produced sample has 5 mM concentration was 2.33 eV. Table (3) summarizes the results of absorption data and energy gap. The results of energy gap showed the Eg decreased with increasing the concentration and corresponding with increasing size of particles in X-ray diffraction, TEM and AFM measurements. The UV-Visible results for produced samples correspond to many previous studies (F. yiing, *et al*., 2015) [27], (A. Rajan, *et al*., 2014) [28].

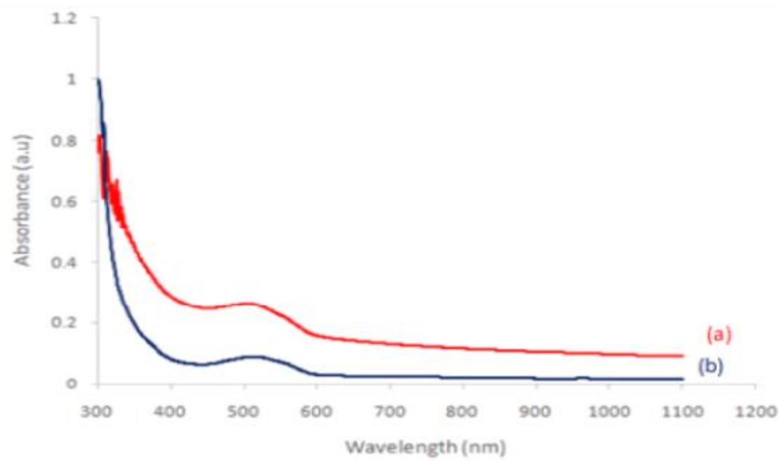


FIGURE 9: UV-Visible absorbance spectra of AuNPs for $HAuCl_4.3H_2O$ with aqueous extract of sumac at concentration, (a) 5 mM and (b) 1 mM.

TABLE 3: Band gaps of the produced samples of gold nanoparticles

Concentration of $HAuCl_4.3H_2O$ (mM)	Absorption wavelength (nm)	Energy gap (eV)
1	503	2.465
5	531	2. 335

CONCLUSION

Gold nanoparticles were synthesized via green method using gold salts ($HAuCl_4.3H_2O$) at two concentrations (1 and 5) mM with sumac aqueous extract as reducing / stabilizing agent with the ratio of mixture 1:1. The presence of gold nanoparticles was confirmed by x-ray diffraction pattern. An average gold nanoparticles size was calculated from XRD, TEM and AFM which increased with increasing concentration of ($HAuCl_4.3H_2O$) with mixture between spherical and cylindrical shape. The SPR was 503 and 531 nm for two concentrations 1mM and 5mM respectively.

Financial and Ethical disclosures

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Conflicts of Interest: Authors declare that they have no conflict of interest.

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