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Green Synthesis of Gold Nanoparticles Reduced and Stabilized by Squaric Acid and Supported on Cellulose Fibers for the Catalytic Reduction of 4-Nitrophenol in Water

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Characterization techniques

UV-Vis spectrum was obtained by using Agilent Cary 50 Conc UV-Visible Spectrophotometer using quartz cuvette of path length 10mm. The Raman analysis was carried out by using Kaiser Optical System RXN1 equipped with Invictus 785 nm diode laser and Andor back illuminated CCD detector with 80 mW laser power for 20 seconds under 40X objective. The Raman intensities were calculated after baseline correction with GRAMS/AI (7.00) software. Dynamic light scattering (DLS) was carried out by using Zetasizer Nano ZS90 with quartz cuvette of 10 mm path length. Zeta potential measurement was carried out using Zetasizer Nano ZS90 with Disposable folded capillary cell. For DLS, Zeta potential and UV-Vis analyses, 6 times diluted SA-AuNPs solution in water was used. Transmission electron microscopy (TEM) experiments were performed using a Hitachi H-7650 transmission electron microscope with an acceleration voltage of 80 kV. Carbon coated copper grids with 200 mesh (Electron Microscopy Science) were used for TEM imaging. 30 microliters of sample were deposited onto the grid and allowed to dry. IMAGEJ software was used to analyze average diameter and size distribution of AuNPs from the TEM images. The results were based on analysis of 86 SA-AuNPs obtained from the TEM image. Energy dispersive X-ray (EDX) analysis was carried out on dried AuNPs, which was centrifuged out at a speed of 15,000 rpm for 30 min. Hitachi S-3400N Type II scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer with accelerating voltage of 15 kV was used to obtain SEM images and EDX spectrum. Glass substrate was used for the EDX analysis of SA-AuNPs, whereas, carbon tape substrate was used for the SEM and EDX analyses of the cellulose fibers supported SA-AuNPs composites. XRPD was carried out using a Bruker D8 Discover XRD using Cu Kα radiation at a scan rate of 2^{o}/min using glass substrate as the sample holder.

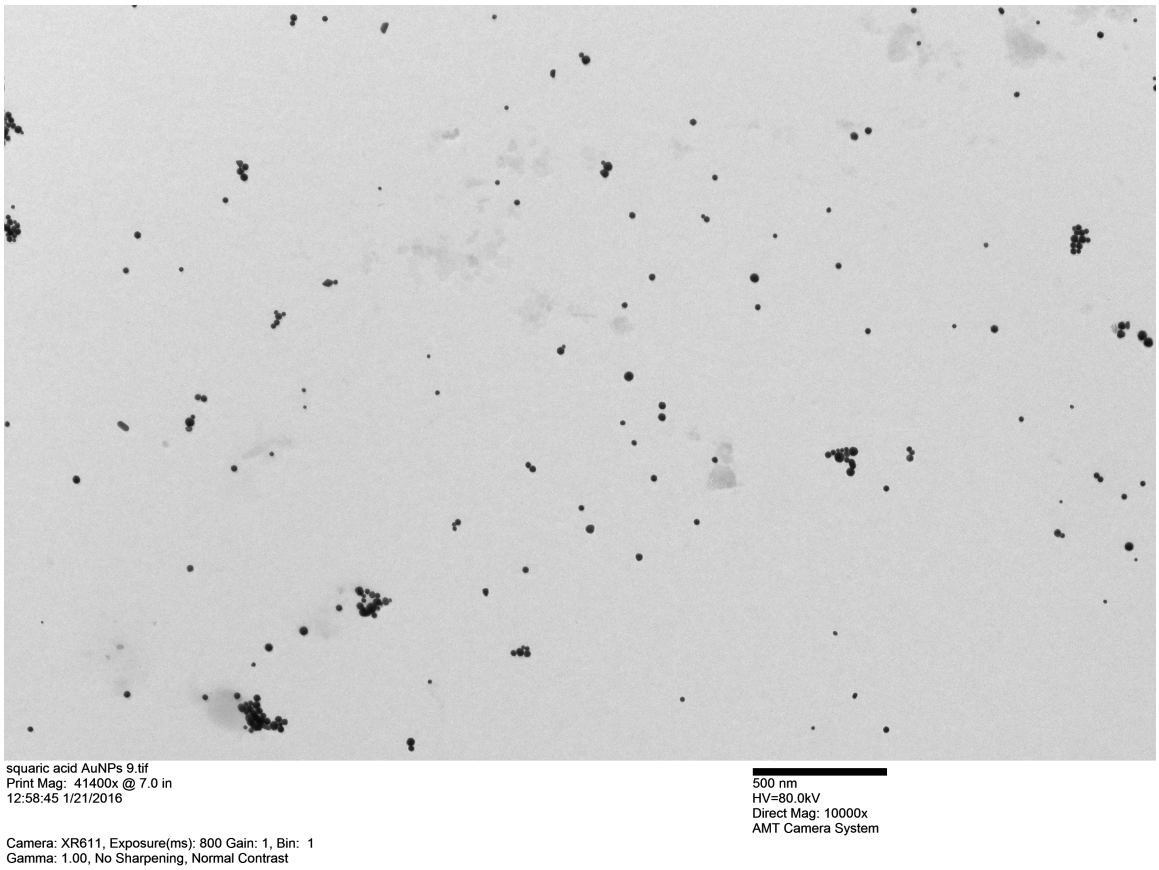

Figure: TEM image of SA-AuNPs

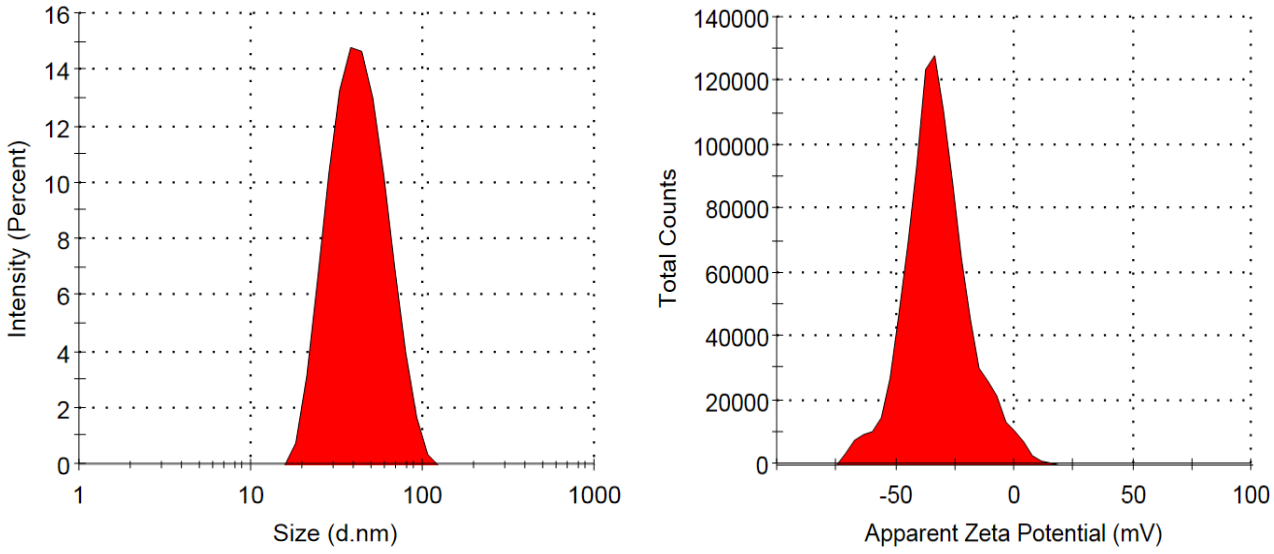

Figure: Dynamic Light scattering and zeta potential measurements of SA-AuNPs in water.

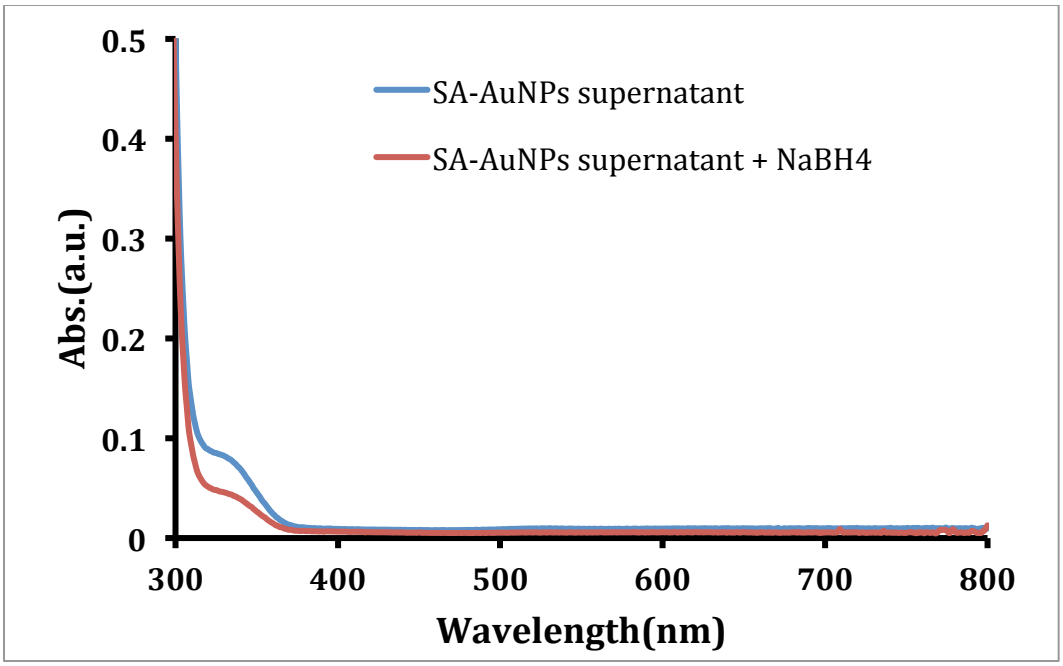

Figure: UV-Vis of the SA-AuNPs supernatant and the SA-AuNPs supernatant with $NaBH_4$.

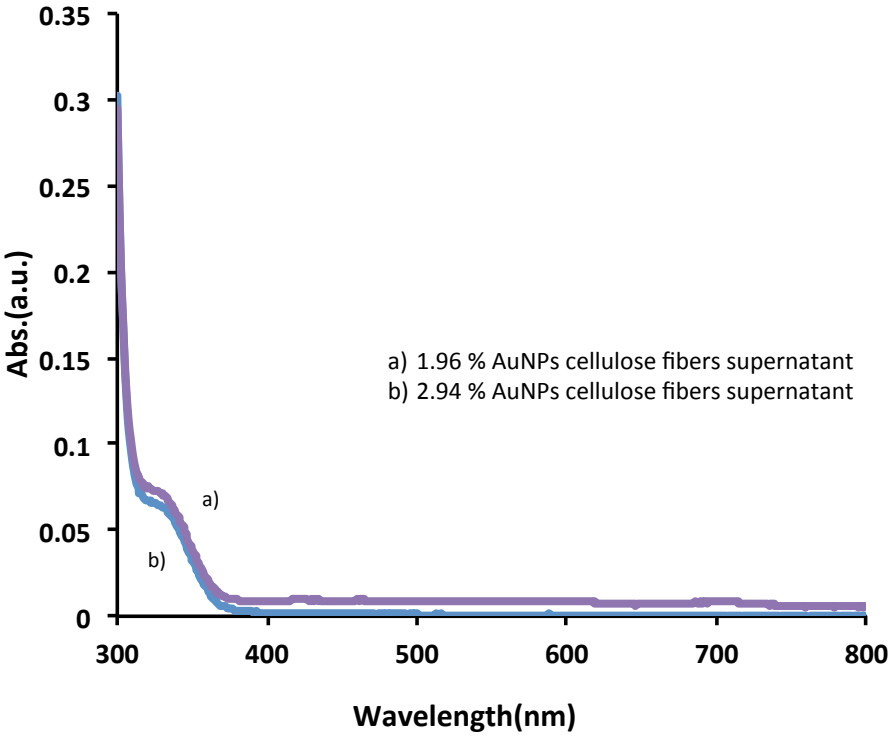

Figure: Uv-Vis of the a) 1.96 % AuNPs cellulose fibers supernatant and b) 2.94 % AuNPs cellulose fibers supernatant

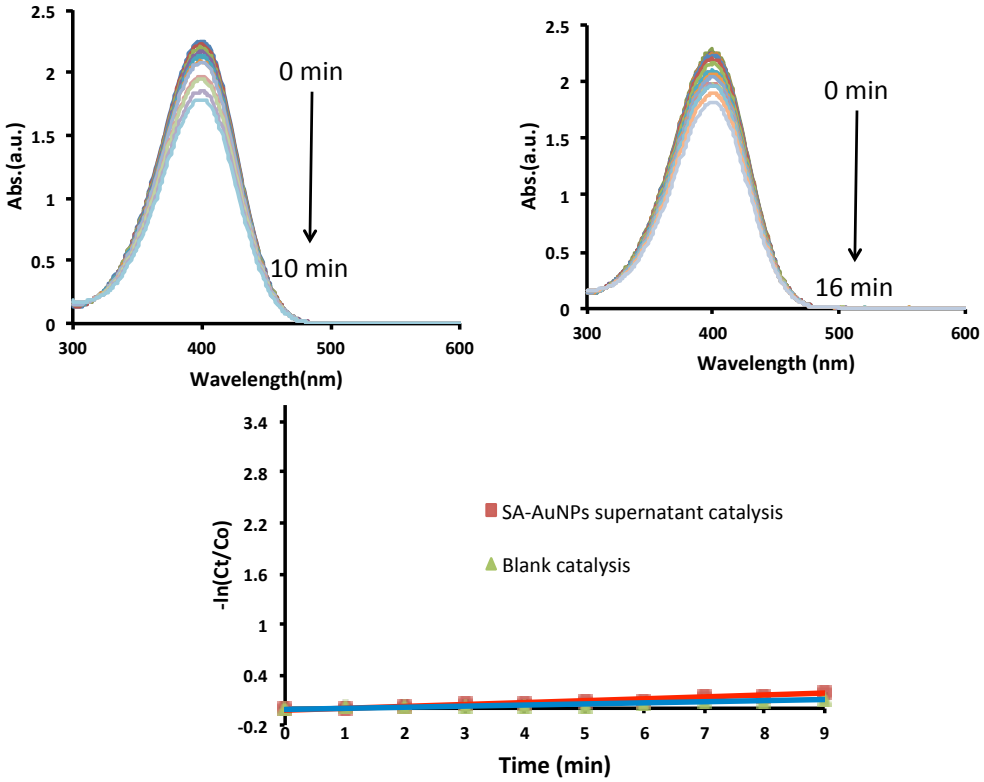

Figure: a) time-dependent UV-Vis of the uncatalyzed e reduction of 4-NP to 4-AP; b) time-dependent UV-Vis of the reduction of 4-NP to 4-AP in the presence of the SA-AuNPs supernatant obtained from ultra centrifugation; c) the corresponding kinetics of the reactions.

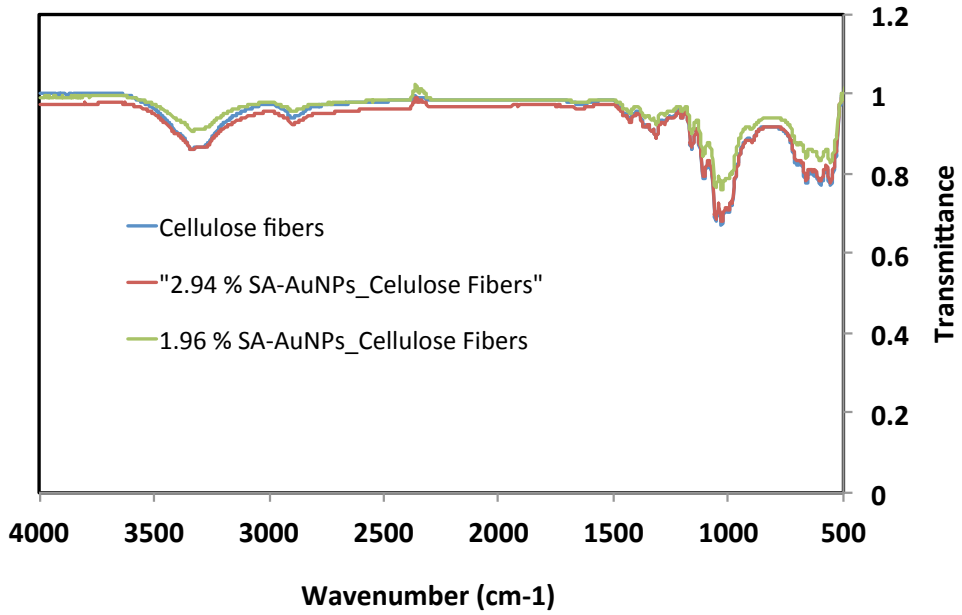

Figure: FTIR spectrum of the cellulose fibers, composite CF-AuNPs-2.94 and CF-AuNPs-1.96.