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# Influence of sodium dodecyl sulfate concentration on the photocatalytic activity and dielectric properties of intercalated sodium dodecyl sulfate into Zn–Cd–Al layered double hydroxide

Abdullah Ahmed Ali Ahmed [a,*], Zainal Abidin Talib [b], Mohd Zobir Hussein [c]

[a] Department of Physics, Faculty of Applied Science, Thamar University, Dhamar 87246, Yemen
[b] Department of Physics, Faculty of Science, Universiti Putra Malaysia, UPM, Serdang, Selangor 43400, Malaysia
[c] Advanced Materials and Nanotechnology Laboratory, Institute of Advanced Technology (ITMA), Universiti Putra Malaysia, UPM, Serdang, Selangor 43400, Malaysia

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ABSTRACT

Sodium dodecyl sulfate (SDS) has been successfully intercalated into Zn–Cd–Al–LDH precursor with different SDS concentrations (0.2, 0.3, 0.4, 0.5 and 1 mol $L^{-1}$) using the coprecipitation method at $(Zn^{2+} + Cd^{2+})/Al^{3+}$ molar ratio of 13 and pH 8. The structural, morphological, texture and composition properties of the synthesized (Zn–Cd–Al–LDH–DS) nanostructure were investigated using powder X-ray diffraction (PXRD), scanning electron microscope (SEM), thermogravimetric analysis (TGA) and Fourier transform infrared (FT-IR), respectively. The photocatalytic activity of these materials was developed by increasing the concentration of intercalated SDS. The absorbance spectra have been used to detect an anion in the LDH interlayer before and after the intercalation process, which confirmed the presence of the dodecyl sulfate ($DS^-$) anion into LDH gallery after intercalation. The anomalous low frequency dispersion (ALFD) has been used to describe the dielectric response of Zn–Cd–Al–LDH–DS nanostructure using the second type of universal power law. At low frequency, the polarization effect of electrodes caused the rising in dielectric constant and loss values. An important result of the dielectric measurements is the calculated dc conductivity values, which are new in dielectric spectroscopy of LDH materials. An important result of the electron spin resonance (ESR) spectra exhibited the successful intercalation of DS molecule into LDH gallery. The *g*-factor value was affected by the SDS concentration which indicated changes to the environment around the DS molecule in LDH interlayer.



## 1. Introduction

Surfactants have been used in the improvement of the removal efficiencies of organic contaminations by the intercalating of these surfactants into interlayer of the layered double hydroxides (LDHs). One of them is sodium dodecyl sulfate (SDS) where its dodecyl sulfate anion ($DS^-$) has been intercalated into different LDH compounds to enhance the sorption of non-ionic organic contaminants [1–4]. The probable configuration of $DS^-$ molecule in the LDH interlayer is perpendicular monolayer and interpenetrating bilayer. These configurations and LDH–DS composition had great impacts on the organic pollutants sorption [5]. The intercalation process to fabricate nanocomposite has been successfully carried out by coprecipitation method [6,7] and ion exchange method [8]. The layered double hydroxide can be described by the general formula of $[M^{2+}_{1-x}M^{3+}_{x}(OH)_2]^{x+}[(A^{n-})_{x/n} \times mH_2O]^{x}$ where $M^{2+}$ is divalent metal, $M^{3+}$ is trivalent metal and $A^{n-}$ is an anion with charge *n*. The value of *m* is the number of mol of water molecules in LDH interlayer per formula weight of compounds [9]. For LDH prepared with ternary cations, its general formula is $[M'^{2+}_{1-x-y}M''^{3+}_{y}M^{3+}_{x}(OH)_2]^{x+}[(A^{n-})_{x/n} \times mH_2O]^{x-}$ where $M'^{2+}$ and $M''^{2+}$ are two different divalent metals and the molar fractions *x* and *y* can be defined as $x = M^{3+}/(M^{3+} + M'^{2} + M''^{2+})$ and $y = M''^{2+}/(M^{3+} + M'^{2} + M''^{2+})$ [10].

Extensive research using the surfactant SDS has been done to enhance the photocatalytic activity and surface area of different structures of the nanomaterials [11–13]. SDS also has been used to prepare different shapes and sizes of $TiO_2$ nanoparticle [12]. Investigation of dielectric properties of SDS in aqueous solution in the frequency range of 30 Hz–6 MHz shows that it is affected by polarization on the surfaces of the electrode (space charge polarization) [14]. The study has also demonstrated that ac

* Corresponding author. Tel.: +967 734125219.
*E-mail address:* abdullah2803@gmail.com (A.A.A. Ahmed).

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electrical conductivities were dependent on the concentration of SDS. Mehrotra and Giannelis [15] used the anomalous low frequency dispersion (ALFD) which can be used to describe the dielectric behavior of the pristine LDH and reasoned that it was due to the presence of two charge carriers; proton of water clusters and nitrate ions in the LDH interlayer. This behavior was also confirmed in our recent studies [16–18]. On the other hand, not much attention has been given to the study of photocatalytic activity and dielectric behavior of intercalated SDS into Zn–Cd–Al–LDH nanostructure.

In the current article, SDS with different concentrations was intercalated to LDH of ternary cations (Zn–Cd–Al–LDH) by the coprecipitation method at a starting molar ratio of $M^{2+}/M^{3+} = 13$ and the pH value of the final slurry was adjusted at 8. The influence of the intercalated SDS on the structural and composition properties of the synthesized (Zn–Cd–Al–LDH–DS) samples was studied. The optical band gap and absorption spectra were obtained. The dielectric properties and electron spin resonance (ESR) spectra for all prepared samples were also investigated.

## 2. Experimental

### 2.1. Materials

The starting chemicals of zinc nitrate ($Zn(NO_3)_2·6H_2O$) (Systerm, 98%), cadmium nitrate ($Cd(NO_3)_2·4H_2O$) (Fluka, 99%), aluminum nitrate ($Al(NO_3)_3·9H_2O$) (Hamburg Chemicals Co., 99.4%), sodium dodecyl sulfate ($C_{12}H_{25}SO_4Na$) (Merck Co., 90%) and sodium hydroxide (NaOH) (Merck Co., 99%) were used without further purification. Deionized water was used as solvent throughout this study, and was boiled before use to eliminate its carbonate molecules.

**Table 1**
Elemental chemical analysis data of Zn–Cd–Al–LDH–DS samples using ICP spectroscopy.

| Sample | Found (wt%) | | | $M^{2+}/M^{3+}$ [a] |
|---|---|---|---|---|
| | Zn | Cd | Al | |
| LDH-0.2 DS | 2.33 | 16.31 | 0.37 | 13.07 |
| LDH-0.3 DS | 2.27 | 16.21 | 0.38 | 12.83 |
| LDH-0.4 DS | 2.35 | 16.80 | 0.37 | 13.42 |
| LDH-0.5 DS | 2.30 | 16.51 | 0.38 | 12.91 |
| LDH-1 DS | 2.26 | 16.28 | 0.37 | 13.15 |

[a] The experimental value of the $M^{2+}/M^{3+}$ molar ratio of samples.

### 2.2. Synthesis of Zn–Cd–Al–LDH–DS

SDS intercalated LDH precursors (Zn–Cd–Al–LDH–DS) with different SDS concentrations (0.2, 0.3, 0.4, 0.5 and 1 mol $L^{-1}$) were synthesized using the coprecipitation method at a starting molar ratio of $M^{2+}/M^{3+} = 13$ and a final pH value of 8. The analysis of elemental percentage in the end products using ICP spectroscopy are presented as shown in Table 1. It also confirmed the starting value of molar ratio of $M^{2+}/M^{3+}$. The synthesis was carried out by a slow addition of three metal nitrates solutions which were $Zn(NO_3)_2·6H_2O$, $Cd(NO_3)_2·4H_2O$ and $Al(NO_3)_3·9H_2O$ with constant stirring. The pH value for all samples was controlled by dropwise addition of aqueous NaOH (0.5 mol $L^{-1}$). The resulting slurry was aged at 70 °C for 18 h in an oil bath shaker (50 rpm). The precipitate

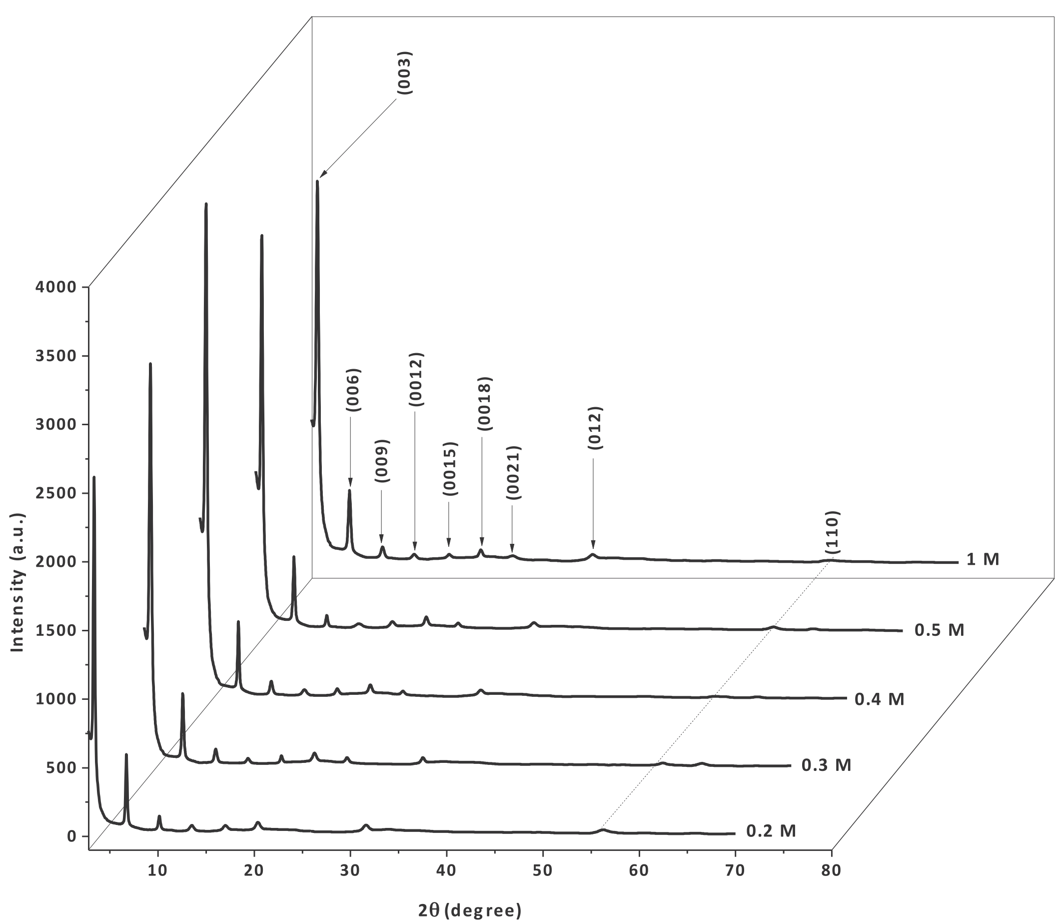


**Fig. 1.** PXRD patterns of Zn–Cd–Al–LDH–DS nanostructure.