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High Performance Humidity Sensor Based on ZnO Nanoparticles Synthesized by Co-precipitation Method

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Abstract. ZnO nanoparticles were successfully synthesized by a low cost co-precipitation method using zinc nitrate and sodium hydroxide as the raw materials. It was observed that the synthesized temperatures greatly effect on the size of ZnO nanoparticles. The lower synthesized temperatures resulted in the smaller nanoparticles. By adjusting the mole ratio of sodium hydroxide, the size of ZnO nanoparticles was also changed. The smallest ZnO particles was 47 nm obtained with 0.7 mole of sodium hydroxide. The smallest ZnO nanoparticles from each synthesized temperatures were fabricated as humidity sensor, showing an impressive performance under different relative humidity (17-94% RH). It should be noticed that the ZnO nanoparticles humidity sensor synthesized at 75 °C exhibited high response 2 times higher than that of synthesized at 95 °C. This is attributed to the higher surface area of ZnO nanoparticles for absorbed water molecule.

Introduction

Humidity sensor is an important device using in industry, biomedicine, automotive, agriculture and also for human comfort. To achieve a high stability, high sensing response, quick response, short recovery time and wide range humidity detection, many types of humidity sensors have been developed over the years. They are surface acoustic wave, capacitive, resistive and optical humidity sensors [1, 2]. Among these, most humidity sensors are either resistive type or capacitive type, and the resistive type humidity sensors showed many advantages, with a good long-term performance, low cost and a processing in mass product over the capacitive type humidity sensors [2].

Currently, ZnO an n-type semiconductor have extensively been investigated since ZnO own many impressive properties. It have been used for variety electronic devices including gas sensor [3], dye-sensitized solar cell [4] and also humidity sensor [5]. It was also reported that ZnO nanostructures humidity sensor exhibited a high stability over a long period test, small drift in hysteresis loops during adsorbed and desorbed water vapor and highly sensing response [5].

In this work, ZnO nanoparticles (*ZNPs*) were synthesized via a co-precipitation method since it is a simple method and does not need an expensive tools. Characterization and humidity sensing property of *ZNPs* was also carried out.

Experimental

ZNPs were synthesized via a low cost co-precipitation method using zinc nitrate ($Zn(NO_3)_2$, 99.9% Sigma-Aldrich) and sodium hydroxide (NaOH, 97% Sigma-Aldrich) as raw materials. Similar to our previous report [6], 0.1 M $Zn(NO_3)_2$ solution and 2 M NaOH solution were separately

prepared using distilled water (DI) as a solvent. $Zn(NO_3)_2$ solution was stirred at 75 and 95 °C for 20 min in a container. NaOH solution was slowly added drop-wise into the container, and mixed solution was successively stirred for 20 min. Then, the mixed solution was allowed to cool down naturally in the air. Precipitated for 24 h, white product in the container was collected, washed by DI water for 4 times, dried in the air for 24 h and annealed at 600 °C for 6 h under air atmosphere to obtain *ZNPs*. The mole ratio between $Zn(NO_3)_2$ and NaOH was set to be 1:0.5, 1:0.6, 1:0.7, 1:0.8 and 1:0.9. *ZNPs* were characterized by scanning electron microscope (SEM), energy dispersive X-ray spectrometer (EDS); JEOL JSM-6335F, X-ray diffractometer (XRD); Rikaku MiniFlex II and Raman spectrometer (Raman); Horiba T64000. Moreover, *ZNPs* were fabricated as humidity sensor and theirs humidity sensing response was investigated under 17-94% relative humidity (RH) by using electrometer, Keithley 6517A.

Results and Discussion

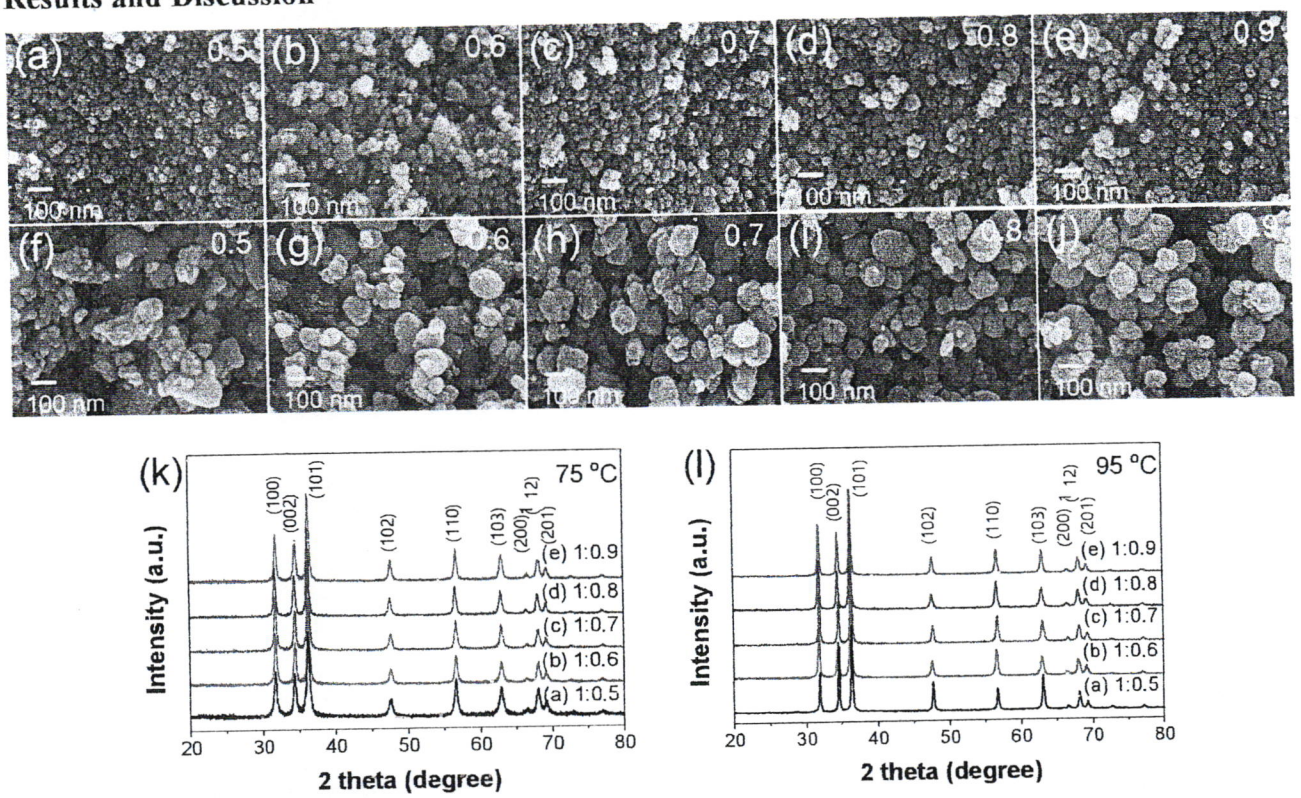

Fig. 1 SEM images of *ZNPs* synthesized at 75 °C (a-e) and 95 °C (f-j) and XRD spectra of *ZNPs* synthesized at 75 °C (k) and at 95 °C (l).

SEM images of *ZNPs* synthesized at 75 and 95 °C were shown in Fig. 1. It was observed that the synthesized temperatures greatly affected on a size of *ZNPs*. *ZNPs* synthesized at 75 °C were smaller than that of 95 °C. By varying NaOH content, the size of *ZNPs* was also adjusted. However, synthesized at 75 °C with 0.7 mole NaOH showed a smallest *ZNPs* (47 nm), Fig. 1(c). This condition also showed the narrowest in size distribution which is the best condition for synthesized *ZNPs* in our work. The average size was observed to be 52, 78, 47, 53 and 47 nm for 75 °C and 92, 107, 99, 90 and 91 nm for 95 °C, respectively. XRD spectra of *ZNPs* were shown in Fig. 1(k) and (l) for synthesized temperatures of 75 and 95 °C. The peaks corresponding to (100), (002), (101), (102), (110), (103), (200), (112) and (201) diffraction planes were detected in the spectra which in accordance with international centre for diffraction data (79-0206) and no impurities peaks were detected. Moreover, the shift of (002) peak (not shown) to higher degree with the increasing of NaOH ratio was observed suggested a change of a lattice parameter c of ZnO crystal [6]. The higher degree the (002) plane shifting the more contract the lattice parameter c.

Chemical composition of synthesized *ZNPs* was examined, and EDS data confirmed that synthesized products were ZnO. The mole ratio of Zn and O atoms was about 1 [3]. Raman technique was performed for crystal defect investigation as shown in Fig. 2(a), for *ZNPs* synthesized at 95 °C. It was observed that peaks observing at 332, 387, 412, 437, 534 and 585 cm^{-1} corresponded to $E_2(H)$-$E_2(L)$, $A_1(TO)$, $E_1(TO)$, $E_2(H)$, $A_1(LO)$ and $E_1(LO)$ vibration modes. No defect or other impurities peaks were detected in the spectra. Similar result was observed for *ZNPs* synthesized at 75 °C (not shown). These data implied that *ZNPs* exhibited a high degree crystallinity with a pure hexagonal wurtzite structure.

Formation of *ZNPs* could be explained as described in ref. [6, 7]. Zinc nitrate solution, acted as the source of Zn^{2+} ions by generated Zn^{2+} and $2NO_3^-$ ions; while alkali salt sodium hydroxide solution supplied OH^- ions by generated Na^+ and OH^- ions when dissolved in DI water. Mixing together leaded a reaction of those and resulted in a formation of $Zn(OH)_2$. By hydrolysis and condensation processes of zinc nitrate with the help of sodium hydroxide in DI medium at low temperatures, *ZNPs* can be formed. Zn^{2+} and OH^- ions played as key factors on growth of *ZNPs* adjusted pH value of the mixed solution together with synthesized temperatures. Though NaOH content impacted the sized of *ZNPs*, influence of synthesized temperature was more pronounced as clearly observed in Fig. 1.

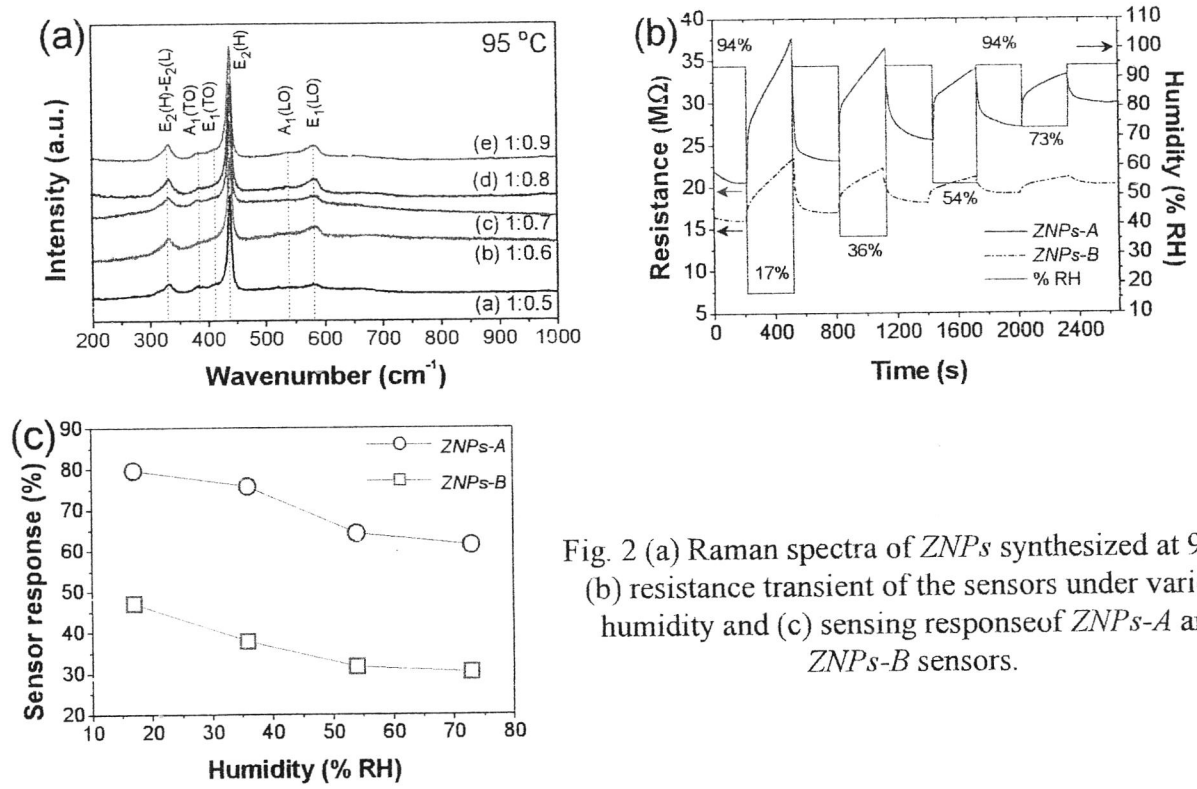

Fig. 2 (a) Raman spectra of *ZNPs* synthesized at 95°C, (b) resistance transient of the sensors under various humidity and (c) sensing responseof *ZNPs-A* and *ZNPs-B* sensors.

The smallest *ZNPs* from both synthesized temperatures were selected and fabricated as humidity sensors since sensor performance directly related to surface area. Sensors fabricated from *ZNPs* synthesized at 75 and 95 °C were denoted as *ZNPs-A* and *ZNPs-B*. The controlled humidity environment was accomplished using 34.2 M NaOH, 16.0 M NaOH, 10 M NaOH, 23.4 M NaCl solutions and DI water for 17, 36, 54, 73 and 94% RH at room temperature. The Resistance transients under various humidity were shown in Fig. 2(b) with 5 V applied voltage. Under high humidity level (94%), both sensor showed a low sensor resistances; however, sensor resistances increased sharply when switched to lower humidity level. This indicated an excellent humidity sensing property of *ZNPs*. The reduction of sensor resistance under humid ambient could be explained as the following. First, the water vapor reacted with ZnO surface by surface collision or self-ionization of water molecules and dissociated to hydroxide ions (OH^-) and protons (H^+). Second, these ionized ions bonded with ZnO lattices via chemisorption process. For more water

vapor, third, water vapor adsorbed on chemisorbed layer via physisorption process. Finally, H^+ ions can move freely in chemisorbed and physisorbed layers which acted as a dominated carrier via proton-hopping mechanism, resulting in a reduction of sensor resistance with the increasing of RH value [5].

The sensing response was carried out using Eq. 1.

$$S = (R_H - R_{94\%}) \times 100\% / R_{94\%} \quad (1)$$

R_H and $R_{94\%}$ are sensor resistances in a given humidity and in 94% RH, respectively [2]. It was noticed that the high sensor performance of *ZNPs-A* was observed, 2 times higher than that of *ZNPs-B* as shown in Fig. 2(c). It was about 80.0, 75.8, 64.3 and 61.4% for *ZNPs-A* and 47.2, 37.9, 32.7 and 30.4% *ZNPs-B* when switched from 94% RH to 17, 36, 54 and 73% RH, respectively. This is attributed to a smaller particle size which provided more grain boundary, more surface area and also more nanopores which offered more active sites for reacting with water molecules [8].

Summary

ZNPs were successfully synthesized using a co-precipitation method. *ZNPs* exhibited a pure hexagonal wurtzite crystal structure. The synthesized temperatures greatly effect on the size of *ZNPs*, whereas NaOH showed a slightly influence. The smallest *ZNPs* from both synthesized temperatures were selected and fabricated as humidity sensors. The sensors showed an impressive performance under different relative humidity (17-94% RH). However, *ZNPs* synthesized at 75°C exhibited a better performance than that of synthesized at 95°C which is attributed to a higher surface-to-volume ratio, providing more active sites for adsorbed water molecules.

Acknowledgment

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17

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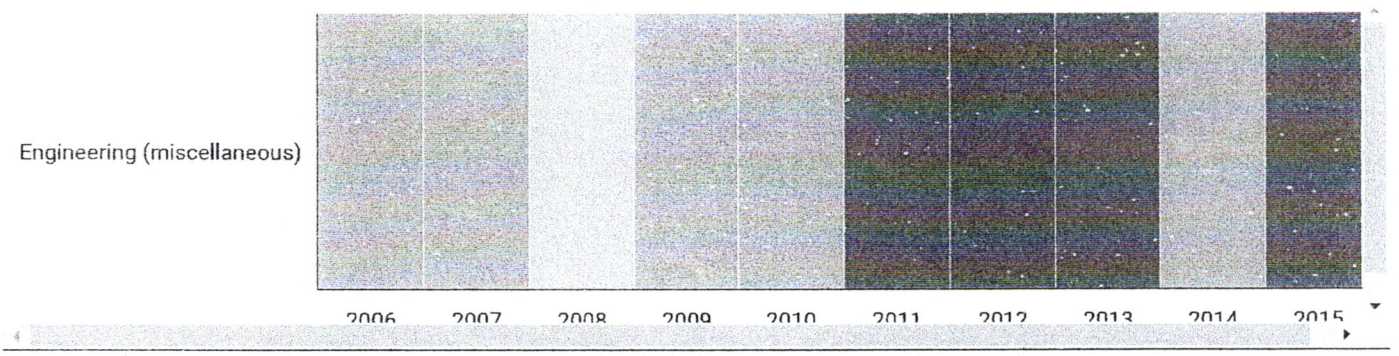

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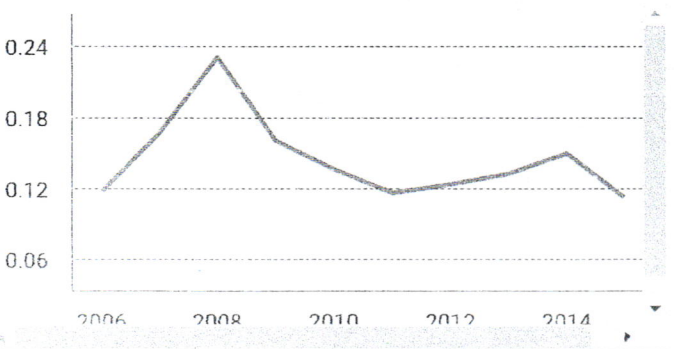

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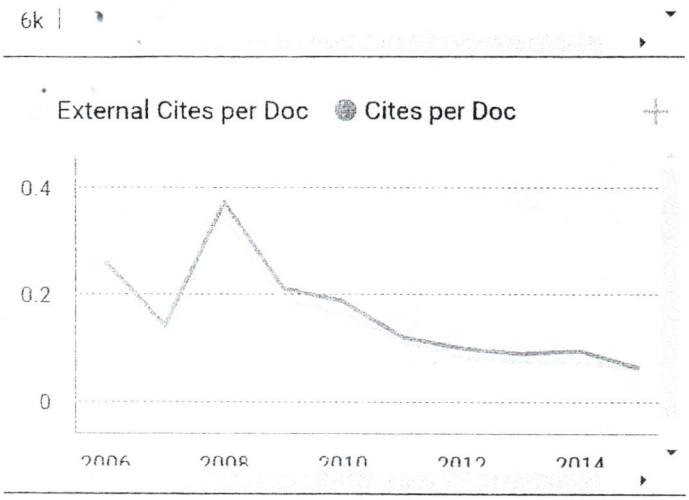

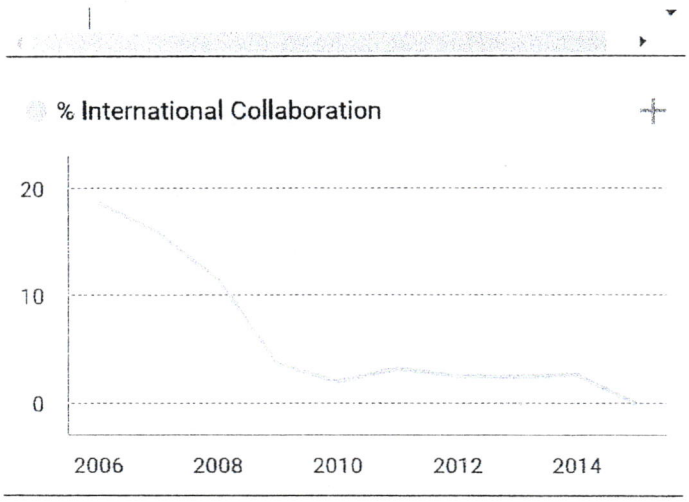

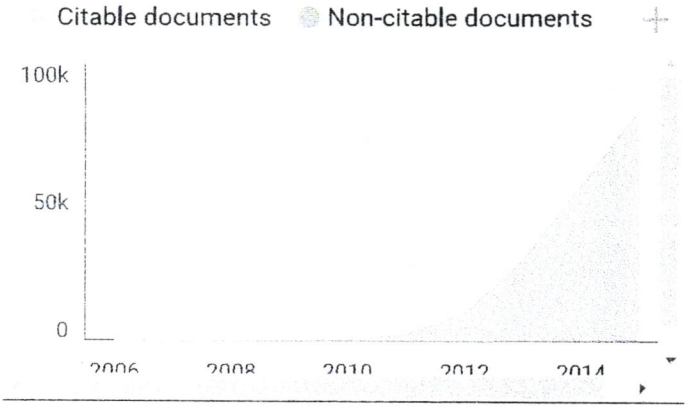

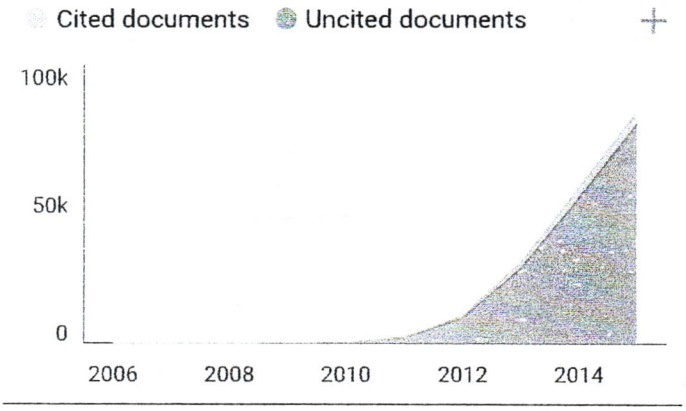

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