Journal of the Taiwan Institute of Chemical Engineers 42 (2011) 868–873

Contents lists available at ScienceDirect

Journal of the Taiwan Institute of Chemical Engineers

journal homepage: www.elsevier.com/locate/jtice

Preparation of nanostructured organic/inorganic polymer hybrids and their humidity sensing properties

Hung-Jie Liou [a], Gobalakrishnan Suyambrakasam [b], Te-Chin Tsai [a], Ren-Jang Wu [a,b,*], Murthy Chavali [a,b,**]

[a] Department of Applied Chemistry, Providence University, Taichung 433, Taiwan
[b] Department of Nanotechnology, School of Interdisciplinary Courses, Noorul Islam Centre for Higher Education (Noorul Islam University), Kumaracoil, 629 180 Tamilnadu, India

ARTICLE INFO

Article history:
Received 17 August 2010
Received in revised form 16 December 2010
Accepted 2 January 2011
Available online 16 March 2011

Keywords:
HPMC
TEOS
Microwave
Nanostructured hybrids
Humidity sensing

ABSTRACT

Humidity sensitive nanostructured hybrid films were synthesized from hydroxyl propyl methyl cellulose (HPMC) and tetra ethoxy ortho silicate (TEOS) via sol–gel route by microwave assistance method. HPMC and TEOS were used as organic and inorganic precursors, respectively. FT-IR spectra of the hybrid material revealed the formation of organic–inorganic networks between HPMC and TEOS. XRD spectra and micrographs showed the formation of HPMC/TEOS amorphous nano hybrids film like structures. Surface roughness was measured using an AFM was 120 nm and measured film thicknesses were between 20 and 24 μm. Hybrid films have showed better linear sensor response, lower hysteresis (<1% RH), faster response (5 s) and recovery time (10 s), and short-term stability (30 min at least). Using Material Studio 3.2 Software, the adsorption phenomena were explained to that of humidity sensing property.

1. Introduction

Humidity is the one of the most important parameter in our environment, especially in storage, preservation and transport operations. Measurement and control of humidity are important not only for human comfort but also for a broad spectrum of industries and technologies. Sensing humidity is extremely significant in agricultural, food, medical and other industrial production fields. For measuring relative humidity (RH) involve infrared hygrometers, aspirated psychrometers and capacitive or resistive type sensors.

Materials that have been studied for humidity sensing include organic polymers (Sakai *et al.*, 1996), ceramics (Cantalini and Perlino, 1992), hybrid polymer/inorganic systems (Su and Tsai, 2004) and composites (Wu *et al.*, 2006), have been investigated and used as humidity sensors, by detecting changes in capacitance or resistance; each of them have their own merits and specific conditions of application (Kulwicki, 1991; Yamazoe and Shimizu, 1986). The RH detection mechanism of polymers has also been based on fluorescence intensity (Zhu *et al.*, 1989), colorimetric absorbance (Zanjanchi and Sohrabnezhad, 2005), SAW devices (Bruno *et al.*, 2004) and optical detection (Bariain *et al.*, 1998) by means of changes in effective refractive index (μ). Most of them show intrinsic shortcomings of instability at high humidity and large hysteresis (Sager *et al.*, 1994), so that they should be modified to improve the sensing characteristics. In the past 20 years, organic–inorganic nanocomposite materials have been regarded as a new class of materials for many new electronic, optic or magnetic applications, since many bulk properties can be improved compared with those of base polymers (Novak, 1993). In need to solve many shortcomings, hybrid material systems, which are one of the most interesting type towards better humidity-sensitive characteristics, such as reliability, ease of processing and low fabrication cost.

Many research groups have demonstrated that, monolithic transparent hybrid materials can be prepared by controlling properly the conditions of hydrolysis and condensation of sol–gel materials such as tetraethoxysilane (TEOS) (Tong *et al.*, 2002) either by bonding chemically or by physical mixing. A significant feature to enhance compatibility in hybrid materials is the formation of covalent bonding between organic–inorganic polymers and inorganic components (Ershad *et al.*, 1997). Hybrid inorganic–organic (nanocomposite) offer a variety of advantageous properties in various applications such as optical materials with high reflective indices, colored glasses, hard coatings, and corrosion protection coatings on the aluminum surfaces, porous materials for chromatography and catalyst supports, and aerogel.

* Corresponding author at: Department of Applied Chemistry, Providence University, 200 Chungchi Road, Shalu, Taichung 433, Taiwan.
Tel.: +886 4 26328001 15212; fax: +886 4 26327554.
** Corresponding author at: Department of Nanotechnology, School of Interdisciplinary Courses, Noorul Islam Centre for Higher Education (Noorul Islam University), Kumaracoil, 629 180 Tamilnadu, India. Tel.: +91 9789 232 232; fax: +91 4651 257 266/250 266.
E-mail addresses: rjwu@pu.edu.tw (R.-J. Wu), ChavaliM@gmail.com (M. Chavali).

1876-1070/$ – see front matter © 2011 Taiwan Institute of Chemical Engineers. Published by Elsevier B.V. All rights reserved.
doi:10.1016/j.jtice.2011.01.001

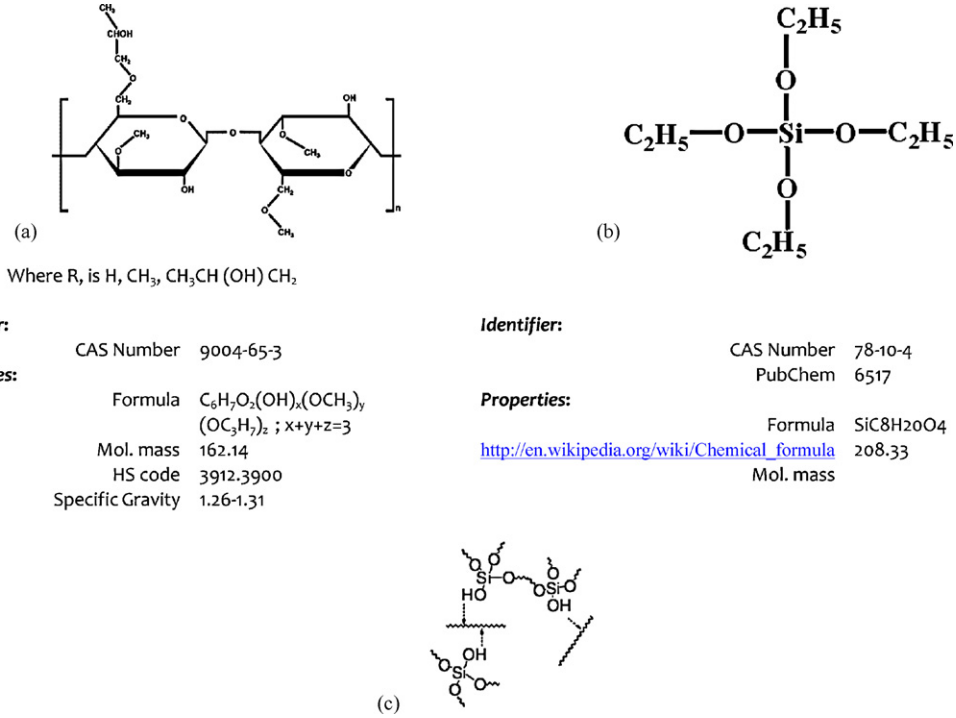

Fig. 1. Structure of (a) hydroxypropyl methylcellulose (HPMC); (b) tetraethyl ortho silicate (TEOS) and (c) HPMC/TEOS hybrid.

Combination of hardness of an inorganic phase and toughness of an organic phase make such systems interesting also from the aspect of mechanical behavior (Chiang *et al.*, 2003).

Hydroxypropyl methylcellulose (HPMC), 2-hydryoxypropyl ether of methyl cellulose is a semi-synthetic ether derivative of cellulose that is widely used in many fields. In particular, it is frequently used in the formulation of controlled release devices because of its non-toxic nature and easy to manufacture (Fu *et al.*, 2004). HPMC belongs to the swellable hydrophilic matrix systems, which form a gel layer when exposed to aqueous media and successfully used in several sensing applications. In this work, we have synthesized and characterized nanostructured organic/inorganic HPMC/TEOS hybrids. These hybrids were investigated for humidity sensing applications. Structures of HPMC and TEOS along with identifiers are shown in Fig. 1. The HPMC/TEOS animated image was shown.

2. Materials and methods

2.1. Chemicals

Hydroxypropyl methylcellulose (HPMC, grade METHOCEL E3 Premium, 3 cps) was a Dow Chemicals product was obtained from Midland, MI, USA, dried in a vacuum dessicator over calcium chloride. Hydrochloric acid (HCl, AR Grade, Merck, Germany) and Tetraethoxy orthosilicate (TEOS) is of analytical grade purity, AR (>99.999%), purchased from Sigma–Aldrich Co., Inc., USA. Water used was distilled and deionized (DI) using a 'Milli-Q' water purification system (Millipore Corp.).

2.2. Sensor preparation

Microwave irradiation of the synthesis scheme (Fig. 2) was done in a domestic microwave oven, a MW73V (Solo MG-TDS 73 V, 20 L Max. Cap. 800 W, Samsung Electronics India Ltd.) was used in this study with availability of 6 power options ranging from very low to very high. Microwave irradiation experiments were performed using a microwave oven equipped with a magnetron (2.45 GHz, 500 W). The reaction was performed in a PTFE beaker at the center of the microwave oven. Optimal parameters used during the microwave assisted synthesis of nanostructured HPMC/TEOS hybrid (MW-HTH) films were given in Table 1. Optimal parameters of films were considered based on their optically transparency, less amount of constituents and homogeneous nature of the films, under microwave irradiation, the dried polymer hybrid films (HTH-5) were obtained within 5 min in comparison with 1 h for conventional

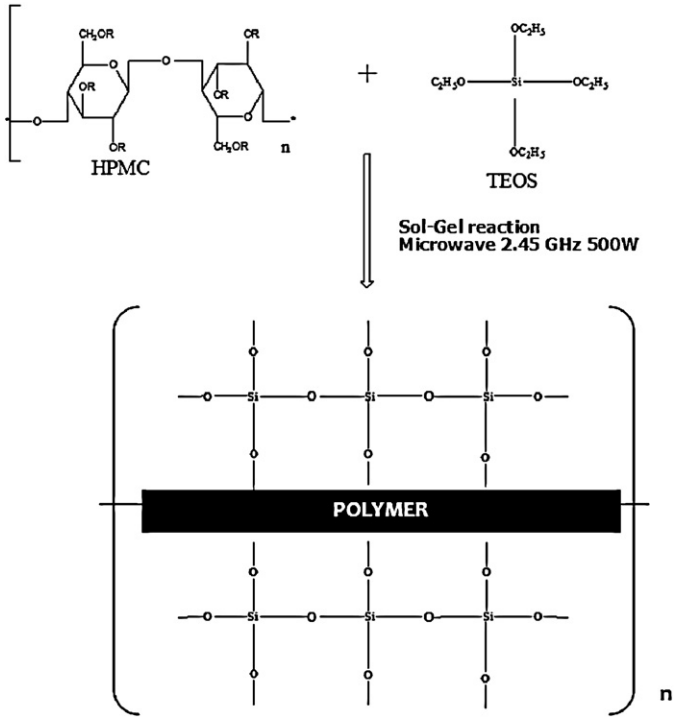

Fig. 2. Microwave assisted synthetic scheme for nanostructured HPMC/TEOS hybrids.