

Preparation and antibacterial efficacy of bamboo charcoal/polyoxometalate biological protective material

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ABSTRACT

Nanocomposites based on Keggin-type polyoxometalate $H_5PV_2Mo_{10}O_{40}$ (POM) and porous bamboo charcoal (BC) were prepared by activation and immobilization processes. The physical properties of the BC/POM composites were examined using FTIR, UV–Vis spectroscope, ^{31}P MAS-NMR, SEM and TEM. These techniques indicated that the POM was intact on the surface of the BC matrix after impregnation. The POM particle size was found to be less than 150 nm based on TEM. The antibacterial effects of the BC/POM composites were assessed from the zone of inhibition, minimum inhibitory concentration (MIC), minimum bactericidal concentration (MBC) and plate-counting method, and an excellent antibacterial performance was discovered.

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1. Introduction

Polyoxometalates (POMs) are oxygen rich class of inorganic cluster systems exhibiting remarkable chemical and physical properties, which have been applied to various fields such as catalysis, materials and medicines [1–5]. POMs perform significant biological activities, with high efficacy and low toxicity. In particular, the sizes and globular structural motifs of many POMs are similar, and in some cases nearly identical to some water-soluble fullerene derivatives showing fairly good anti-HIV activity (human immunodeficiency virus) [6–8]. So far, various biological effects of the POMs have been reported on antitumor and antiviral activities [9,10]. With regard to the antibacterial activity of the POMs, we found that the polyoxometalates with Keggin, lacunary Keggin, Wells-Dawson, double-Keggin and Keggin-sandwich structures enhanced the antibacterial activity of β -lactam antibiotics on methicillin-resistant *Staphylococcus aureus* (MRSA) [11], *Helicobacter pylori* [12] and SARS coronavirus (SARS-CoV) [13].

In practice, the applications of POMs as solid phase catalysts have been limited by their low surface areas (1–10 m²/g) and their low decomposition temperatures. A number of attempts have been made to disperse POMs on inert supports, with the goal of effectively increasing their surface areas and hence the number of accessible active catalytic sites. Several materials such

as carbon [14], zirconia [15], titania [16], alumina [17] and porous silica [18,19] have been used as solid supports for the dispersion of POMs. To find a suitable support able to attach itself firmly and to disperse the POMs would be of great practical interest because catalytic activity will be related to the higher surface area and to the thermal stability of the supported heteropolyanion.

Bamboo charcoal has a number of beneficial characteristics, including high electric conductivity and self-lubricity, and can be used as a friction material and an electromagnetic shield material [20]. On the other hand, an increasing number of bioresources have also been utilized in the preparation of adsorbents, and some studies have shown that certain bioresources have high potential for use as adsorbents. Among these, bamboo is recognized as one of the most popular bioresources, and its adsorption characteristics have been the subject of many studies [21,22]. In our research work, we try to combine $H_5PV_2Mo_{10}O_{40}$ with bamboo charcoal, aiming to explore a kind of water-insoluble and biocompatible antimicrobials. In order to explore the antibacterial effects of BC/POM composites, zone of inhibition testing, minimum inhibitory concentrations (MICs), minimum bactericidal concentration (MBC) and the plate-counting method were used in this study to examine the antibacterial activity of the BC/POM composites against gram-negative *Pseudomonas aeruginosa* (*P. aeruginosa*), Ciprofloxacin-resistant *P. aeruginosa* (CRPA), *Escherichia coli* (*E. coli*) and *E. coli* JM109 (pUK18), and gram-positive *Staphylococcus aureus* (*S. aureus*), Methicillin-resistant *S. aureus* (MRSA) and *Bacillus subtilis* (*B. subtilis*).

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2. Experimental

2.1. Preparation of BC/POM composites

BC powders (particle size $<10\ \mu\text{m}$, Taiwan Paiho) were activated with nitric acid solution under stirring for 1 h. The activated BC powders (5.0 g) were then immersed in 60 ml of ethylacetate under N_2 atmosphere and heating at $60\ ^\circ\text{C}$ for 3 h. Polyoxometalate ($\text{H}_5\text{PV}_2\text{Mo}_{10}\text{O}_{40}$) was purchased from Japanese Inorganic Chemistry Industry Joint-stock Company and was used as received. POM loading of about 1.25, 2.5, 5.0, 10.0 and 15.0 g was dissolved in 60 ml of ethylacetate and added the resulting solutions to the above mixture. Stirring was continued under an inert atmosphere at room temperature for another 3 h. The BC/POM particles were separated and washed with ethanol, then dried in a vacuum at $100\ ^\circ\text{C}$ overnight. The weight ratios of BC:POM prepared were approximately equal to 1:0.25, 1:0.5, 1:1, 1:2 and 1:3. These samples were referred to as BC/POM-0.25, BC/POM-0.5, BC/POM-1, BC/POM-2, and BC/POM-3, respectively.

2.2. Characterization

FTIR and UV-vis spectra of the samples were recorded on a Tensor 27 (Bruker) and an UV-3000 spectrophotometer. ^{31}P MAS-NMR spectra were recorded on a Bruker DSX 400WB solid state NMR spectrometer at a resonance frequency of 400 MHz. The morphology of the composites was observed using a scanning electron microscope (SEM, Hitachi S-800) and a transmission electron microscope (TEM, Philips CM-200) equipped with an energy-dispersive X-ray (EDX, Hitachi S-300) microanalysis system. The Brunauer-Emmett-Teller (BET) specific surface areas (S_{BET}) of the BC/POM composites were determined by a NOVA 1000e automatic physical absorber using highly purified nitrogen gas at 77 K.

2.3. Test of antibacterial properties

P. aeruginosa (ATCC 27853), CRPA, *E. coli* (ATCC 25922), *E. coli* JM109, *S. aureus* (ATCC 25923), MRSA and *B. subtilis* were obtained from the Food Industry Research and Development Institute, Taiwan, and were used as the reference strains in antibacterial testing. The antibacterial spectrum of BC/POM composites was evaluated by zone of inhibition test. A standard inoculum of the test organism with 10^7 colony-forming units (CFU)/ml was swabbed onto the surface of a Muller-Hinton agar plate, and then discs of filter paper impregnated with antibacterial agents (6 mg/ml) were placed on the agar. The plates were incubated overnight at $37\ ^\circ\text{C}$, and the clear zones around the disc were measured.

The antibacterial effects of the composites were evaluated by the determination of minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC) using the broth dilution method. Tubes containing 3 ml MH broth with 10-fold dilutions of the BC/POM composites ranging from 4 mg/l to 256 mg/l were inoculated with 10^7 CFU/ml of bacteria. The inoculated tubes were then incubated at $37\ ^\circ\text{C}$ for 18 h. After incubation, tubes were examined without shaking for visible turbidity; the MIC was determined as the lowest dilution of composites that produced no visible turbidity [15]. The test was performed three times for each strain, and results in agreement on two or more occasions were adopted as the MIC of the strain. After the MIC has been determined, 0.1 ml of inoculum from each of the tubes broth without visible turbidity was subcultured on nutrient agar plate and incubated at $37\ ^\circ\text{C}$ for 24 h. The number of grown colonies on this subculture after incubation period was counted and compared to the number of CFU/ml in the original inoculum. The lowest concentration of BC/POM composites that allowed less than 0.1% of the original inoculum to survive, was said to be the MBC [23].

To further investigate the antibacterial effects of the composites, the plate-counting method was used [24]. Approximately 10^7 CFU of *S. aureus* were cultured on MH agar plates supplemented with BC/POM composites. BC/POM-free MH plates cultured under the same conditions were used as a control. The plates were incubated at $37\ ^\circ\text{C}$ for 18 h and the numbers of colonies were counted. The test process was described as follows: 60 mg of the BC/POM composites were added into 3 ml of MH broth containing 10^7 CFU/ml bacteria. The mixture was aerobically incubated at $37\ ^\circ\text{C}$ under vibration for 24 h. The above suspension (30 μl) was cultured on an agar plate and incubated at $37\ ^\circ\text{C}$ for 18 h.

3. Results and discussion

3.1. Structure characterization

Fig. 1 shows the FTIR spectra of pure POM and BC/POM samples from 400 to $1400\ \text{cm}^{-1}$. The band at about $1062\ \text{cm}^{-1}$ of POM is attributed to the $\nu_{\text{as}}(\text{P}-\text{O}_i)$ vibrations, while the bands at about 962, 865 and $780\ \text{cm}^{-1}$ are generally assigned to stretching vibrations due to $(\text{M}-\text{O}_t)$, $(\text{M}-\text{O}_c-\text{M})$ and $(\text{M}-\text{O}_e-\text{M})$ species ($\text{M} = \text{Mo}$ or V) [25]. It has been reported that when POM is bound to a support, a shift in the band position due to the Keggin complex is observed, resulting from a change in the chemical environment and the number and types of counterions [26]. As shown in Fig. 1, BC/POM samples, the absorption bands assigned to the $\text{M}-\text{O}_c-\text{M}$ and $\text{M}-\text{O}_e-\text{M}$ stretching at 865 and $780\ \text{cm}^{-1}$, respectively, are shifted to 873 and $800\ \text{cm}^{-1}$. These results indicate that there were interactions between the BC particles and the POM. Fig. 2 shows the UV-vis spectra of the POM and BC/POM composites. The UV-vis spectra of POM and BC/POM composites dissolved in solution show prominent bands at 217 and 309 nm, its intensity distinctly increased with increasing POM content. These bands are generally assigned to charge transfer from the bridging oxygen to the metal in polyoxometalates [27].

^{31}P MAS-NMR is a useful tool in detecting the local environments of POM, since the chemical shift of the phosphorous atom depends not only on its local environment within the metal cluster but also on factors such as associated water molecules, metal ions and solid supports [28]. Fig. 3 shows the ^{31}P MAS-NMR spectra of unsupported bulk POM and BC/POM composites. All the samples displayed more than one phosphorus resonance, which indicates that the structure is complex. Bulk POM exhibited a sharp and in-

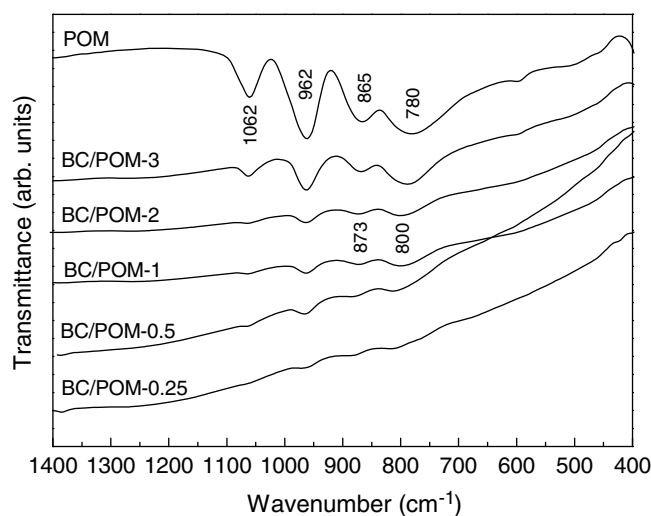


Fig. 1. FTIR spectra of POM and BC/POM composites.

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