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Research Article

Cellulose-Based Polymer Composite with Carbon Black for Tetrahydrofuran Sensing

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This work focused on studying the sensing efficiency of tetrahydrofuran (THF) by composite films made of thin layers of a cellulose-based polymer and carbon black. We analyze the reproducibility, durability, desorption time, and the sensitivity percent as a function of the amount of solvent. Two types of experiments were conducted, (1) progressive sensing test (PST) which consisted of progressively increasing the amount of solvent from 0.1 mL increments up to 1.0 mL and (2) multiple sensing test (MST) where the layers were subjected to consecutive pulses of the same amount of solvent, with a minimum of 0.1 mL and a maximum of 0.4 mL. The response and desorption times were a few seconds, and the sensitivity percent ranged from 1% to 170% and was dependent on the solvent quantity.

1. Introduction

The polymer/carbon particles composites using carbon black (CB) and carbon nanotubes (CNT) have been extensively investigated for detecting an array of solvent vapors [1–20]. These materials function at room temperature, and it is possible to detect a wide variety of compounds through the appropriate choice of polymer and conductive particles.

The most critical characteristics of these compounds, for use in sensors, are the sensibility, response and desorption times, and durability. Controlling of these parameters in polymer matrices made from renewable sources is of great interest, and there are many potential applications for use in vapor sensing devices.

Cellulose is the most abundant natural product, in addition to being biodegradable and renewable. It is a colorless polymer, insoluble in water, dilute acids and alkalis and in the most of organic solvents. In addition, cellulose can, via esterification and etherification reactions in pendant hydroxyl groups, yield a wide variety of derivates. Cellulose esters and ethers can be easily dissolved in aqueous and/or organic solvents. Acrylamidomethyl cellulose acetate butyrate (ACAB) is an ester derived from cellulose obtained by reacting cellulose acetate butyrate (CAB) with N-methylacrylamide (NMA) in an aqueous phase and in the presence of sulfuric acid catalyst. The product, a white powder, is used to produce copolymers with optimum processing properties [21] used in the fabrication of composite materials. Figure 1 shows the chemical structure and composition of ACAB.

In a previous paper [21], we studied the electrical properties of ACAB composites with carbon black. The composites exhibited a critical concentration of 3 wt.% CB. This property and ease of processing make ACAB an attractive material for sensing organic solvent vapors. In this work, we studied

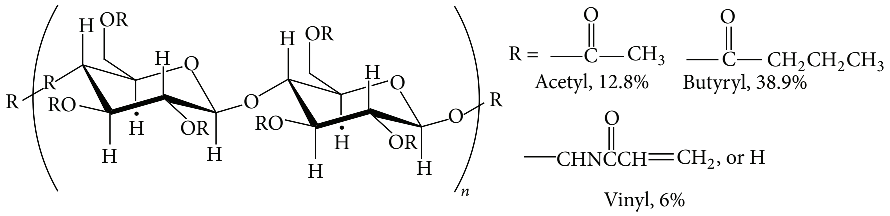


Figure 1: Structural representation and composition of commercial ACAB.

the THF vapor detection efficiency using thin layers of an ACAB/10 wt.% CB composite. Sensitivity percent, response, relaxation times, and the durability were studied as a function of the amount of solvent. These parameters were evaluated in two types of experiments: (1) in the first experiment, progressive sensing test (PST), samples were subjected to pulses in which the amount of solvent was progressively increasing from 0.1 mL in 0.1 mL up to complete 1 mL. This methodology was useful in determining the electrical behavior of the composite as a function of solvent level. This testing also served to establish the minimum and maximum limits of detection in the range of 0.1 to 1.0 mL. (2) The second type of experiment, multiple sensing testing (MST), consisted of subjecting the composite layers to the same amount of solvent repeatedly until deterioration ensued. The fixed amounts established for this second experiment (0.1 and 0.4 mL) were determined in accordance with the reproducibility of the results obtained in the PST experiment. Sensing experiments were carried out in an in-house designed chamber. The sample was placed in the chamber, conditioned via a flow of moisture-free air, and then exposed to controlled solvent amounts while the electrical resistance was monitored.

2. Experimental Part

2.1. Reactants. Acrylamidomethyl cellulose acetate butyrate (ACAB) was obtained from Sigma-Aldrich, Chemical Company. The number-average molecular weight, M_n is about 10.000 as determined by GPC. The material is soluble in a variety of acrylic monomer. Other properties include: density of 1.31 g/mL at 25°C, viscosity of 60 cp at 25°C, T_g of 118°C, and T_m of 146°C. Carbon Black Vulcan XC72 was donated by Cabot Co. The particle size is in the range of 32 nm, and conductivity is 10–10^2 S/m. HPLC grade tetrahydrofuran (THF) was obtained from Sigma-Aldrich, Chemical Company Inc. It is a solvent with medium polarity that allows an optimum dispersion of carbon black [21–23] and dissolves ACAB. The molecular weight is 72.11 g/mol, the bp 66°C, vapor pressure 19.3 kPa at 20°C, and density 0.88 g/cm^3. Silver Paint was provided by Electron Microscopy Sciences. It is a colloidal silver liquid with 72% in solids and a contact resistance of 1 Ω.

2.2. Preparation of the Polymer Composites. The polymer composites were prepared in accordance with the methodology reported in [21]. Layers of polymer composite were deposited on glass substrates of 2.5 × 2.5 cm, at a speed of 1000 rpm in a spin coating system. Each layer of polymer was obtained by 10 consecutive deposits of ACAB/10 wt.% CB dissolution, leaving a time of approximately 3 min between each deposit.

The layers were put in a vacuum for 24 hr in order to remove the residual solvent. Electrical contacts were put on the layer composite by painting two parallel lines of silver paint with a distance of 1.5 cm of separation. The layers were selected in accordance with the range of their electrical resistance (a few MΩ in order of magnitude) for each group of experiments.

2.3. Sensing Tests. THF vapors were detected using a system in which the electrical resistance of the layer is monitored with a multimeter STEREN MUL model 600 coupled to a computer. The layer first is subjected to a flow of moisture-free air and oil at a rate of 1 L/min. After that, the air is infused with controlled amounts of THF pulses. Each pulse is applied by injecting the respective amount of THF using graduate syringes. Between pulse and pulse, a 10 to 15 min is left so that the resistance returns to a stable value. The layer in relation to the air flow is always placed at a right angle as shown in Figure 2.

2.3.1. Progressive Sensing Test (PST). In this type of experiment, the layers were exposed to a progressive increasing of THF pulses in 5 samples with the same range of resistance, from 1 to 2 MΩ. Pulses were applied from 0.1 to 1 mL with increments of 0.1 mL. After the completion of all the pulses, the sample is put in a vacuum for 24 hr to remove the residual solvent, and again it is exposed to the same process. This cycle is made repeatedly (5 times) in order to get a statistics.

2.3.2. Multiple Sensing Test (MST). This experiment used layers with the same range of resistance, from 2 to 3.5 MΩ for multiple sensing using 0.1 mL of solvent, and from 5 to 6.5 MΩ for using 0.4 mL. Layers were subjected to repetitive pulses of the same amount of THF until deterioration of the sample was detected. In all cases, this deterioration was identified by an interruption of the electrical signal or because the resistance values were not reproducible, outside of the average of the previous measurements.

3. Results and Discussion

3.1. Tests Progressive Sensing. The initial characteristics of the used samples are shown in Table 1.

Figure 3 illustrates a typical PST experiment. Each signal is produced in response to a pulse of solvent in quantities that increment from 0.1 to 0.1 up to 1 mL, starting with 0.1 mL.

Table 1: Original resistance (R_0) for the layers used in each test before any THF pulse.

For PST		For MST at 0.1 mL		For MST at 0.4 mL	
Sample	R_0 (MΩ)	Sample	R_0 (MΩ)	Sample	R_0 (MΩ)
PST 1	1.637	MST 1a	3.539	MST 1b	5.499
PST 2	1.181	MST 2a	3.328	MST 2b	6.190
PST 3	1.553	MST 3a	3.090	MST 3b	6.559
PST 4	1.108	MST 4a	2.109	MST 4b	6.288
PST 5	1.765	MST 5a	4.44	MST 5b	6.190

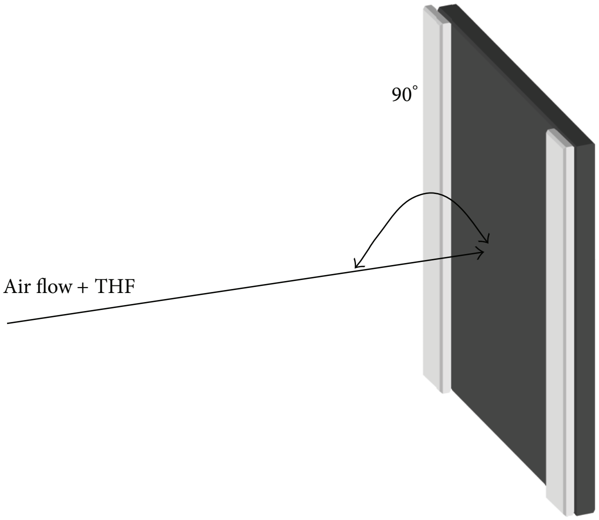


Figure 2: Experimental arrangement for sensing test.

The sensitivity percent, response, and desorption times were determined according to the definitions given in [24]. The resistance after a pulse never returns the R_0 value, being R_0 the original resistance before any pulse. The sensitivity of each pulse was calculated using the R_i value corresponding to the value at which the resistance returns from the previous pulse. Figure 3 shows how R_i value increases as the amount of solvent increases, and this is usually higher than the initial R_0 value. This effect could be the result of the fact that some solvent is retained by the layer and/or there is a modification of the initial configuration of the conductive networks derived from the swelling of the polymer matrix in contact with the solvent. A similar effect takes place in thin layers of polymer composites when they are exposed to several cycles of heat treatment [23]. The average value of the sensitivity percent is shown in Table 2.

As the amount of solvent increases, the detection sensitivity percent also increases. For a pulse of 1 mL, the sensitivity percent is nearly 180 times greater than the sensitivity for a pulse of 0.1 mL. In the PST experiment, the increase in sensitivity is almost linear between pulse and pulse, from 0.1 to 0.4 mL. The percent of higher sensitivity is presented for THF volumes from 0.5 to 1.0 mL; however, the error in the changes of sensitivity is higher than for volumes from 0.1 to 0.4 mL. For all the THF volumes, the sensitivity percent is greater than the unit, which is an indication, as mentioned before, that the compound of ACAB with CB is a sensitive sensor to THF.

Table 2: Sensitivity average (SA), error percent, and response time for the progressive sensing tests.

THF pulse (mL)	SA (%)	Error (%)	t_R (s)
0.1	1.53	±1.34	11
0.2	2.10	±3.32	13
0.3	4.50	±22.23	13
0.4	15.70	±98.21	24
0.5	33.93	±43.00	11
0.6	57.65	±11.45	15
0.7	105.83	±37.42	9
0.8	131.95	±33.74	8
0.9	143.78	±7.58	4
1	178.83	±17.68	2

The response time is defined as the time that it takes until the detector reaches the 90% of the maximum resistance [24]. The average of the response time in the PST experiments is shown in Table 2. In this type of experiments, the response times of the ACAB with CB composite to THF in the range of 0.1–1.0 mL take no longer than 24 s, and this is very good for sensors derived from polymeric composites. The response times have a tendency to decrease with the amount of THF. This indicates that THF has to be absorbed via diffusion and swelling of the polymer matrix. This swelling produces a separation of the conductive particles and, in turn, a variation

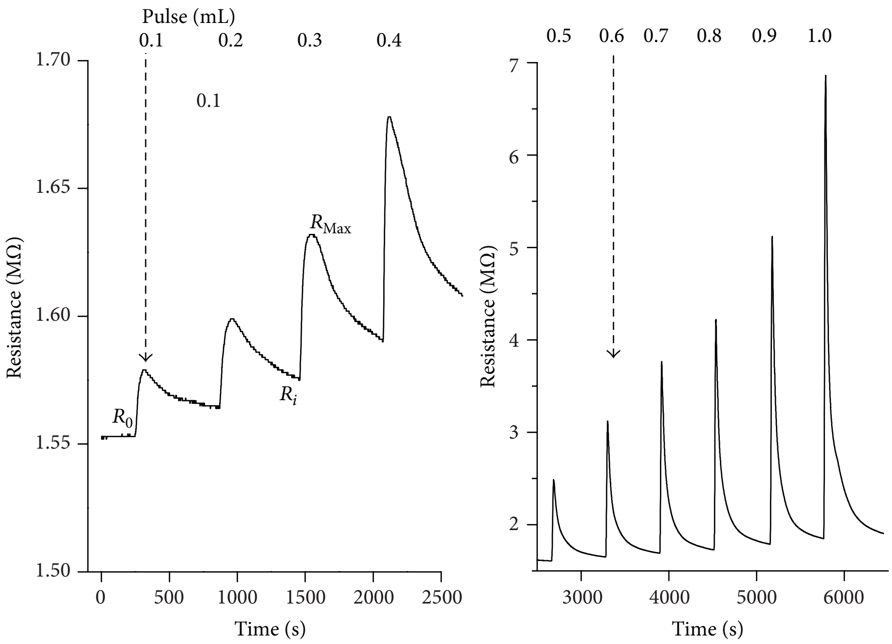


Figure 3: Progressive sensing test from 0.1 to 1 mL of THF for PST 3 sample.

in the electrical response or resistance. With a small amount of solvent, there is a slight penetration of the surface of the layer, and the change in resistance is not significant. As the amount of solvent increases, absorption and swelling are greater, and the response to the detection is incremental. In this case, the response is detected in the first few seconds that the composite material comes in contact with the solvent.

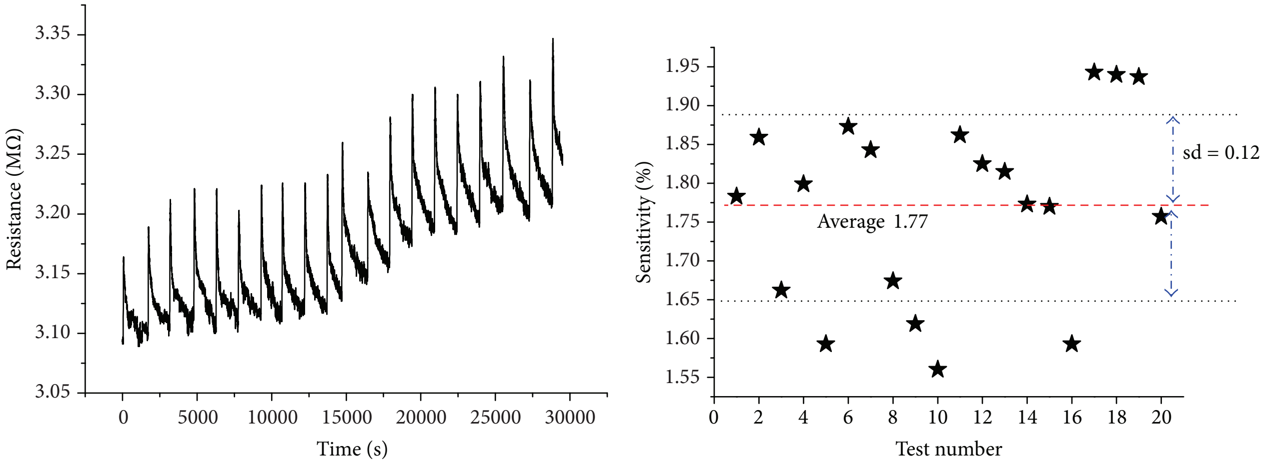
The desorption time is the time taken for the layer composite to release the absorbed solvent at a specific resistance value. This value corresponds to that at which the resistance has reached the 10% of its saturation value after it has decreased from the maximum value [24]. It is noticed that as the solvent amount increases there is a reduction in desorption times. A part of the solvent remains absorbed in the layer (as evidenced by the greater initial resistance in each subsequent pulse). When low amounts of solvent are used, most remains absorbed by the layer and desorption of minute amounts of it is a very slow process. For higher solvent amounts, some vapor also persist but the rest is desorbed immediately, making the desorption process faster.

3.2. Multiple Sensing Test (MST). The purpose of this experiment was to evaluate the reproducibility of the composite by applying a consecutive series of pulses using the same THF quantity. 0.1 and 0.4 mL quantities of THF were studied. The values of initial resistance for the composite layers are shown in Table 1. The results of the multiple sensing tests are shown in Figure 4(a). Each peak represents the response to an injected pulse of 0.1 mL THF. The average of the sensitivity percent of five samples subjected to 20 pulses of 0.1 mL in this MST experiment is displayed in Table 3.

The sensitivity percent is shown in Figure 4(b), and it is compared with the average value that is 1.77. The percent error with respect to the media is ±5.64%. Thirty-five percent are outside of the range of error; therefore, 65% of the pulses are accepted as the reproducibility sensing percent, with a continuous average service of 9.3 hr for each layer, equivalent to 20 pulses. In the same way as in the previous test, five layers were evaluated for pulses of 0.4 mL. The same methodology

Table 3: Sensitivity average for the MST experiments to 0.1 and 0.4 mL THF.

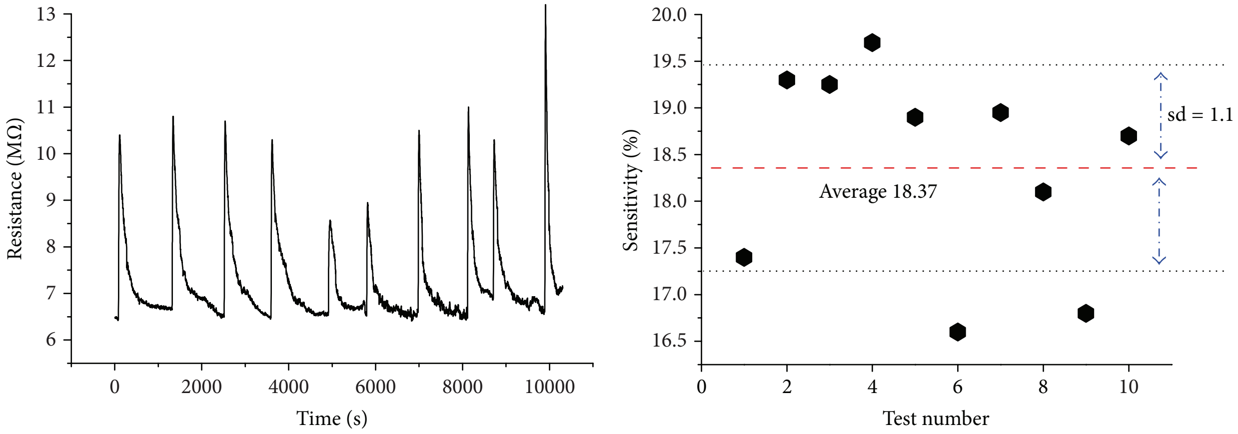
Test number	SA (%) for 0.1 mL	SA (%) for 0.4 mL
1	1,783	17.400
2	1,859	19.300
3	1,662	19.250
4	1,799	19.700
5	1,593	18.900
6	1,873	16.600
7	1,843	18.950
8	1,674	18.100
9	1,619	16.800
10	1,560	18.700
11	1,862	—
12	1,825	—
13	1,815	—
14	1,773	—
15	1,770	—
16	1,593	—
17	1,943	—
18	1,940	—
19	1,937	—
20	1,757	—
Average	1,774	18.370



(a)

(b)

Figure 4: For the MST-3a sample: (a) MST experiment to 0.1 mL THF and (b) reproducibility to the MST experiment at 0.1 mL THF.



(a)

(b)

Figure 5: For the MST-3c sample: (a) MST experiment to 0.4 mL THF and (b) reproducibility to the MST experiment at 0.4 mL of THF for the same sample.

was used to evaluate five layers with pulses of 0.4 mL. The original resistance of the layers is shown in Table 1.

A multiple sensing test using 0.4 mL THF is shown in Figure 5(a). It is noted that the sensitivity is higher for pulses of 0.4 mL than for 0.1 mL; however, at high volumes the deterioration of the sensor layer is faster. This is detected by the lower number of pulses of 0.4 mL THF in which the layer senses before it is damaged. The average sensitivity of the five evaluated layers is shown in Table 3. The number of performed tests was, on average, 10 pulses with an error of ±5.08% in comparison with the average value (18.37%). The results are shown in Figure 5(b), where the average sensitivity is the dotted line; the solid lines represent the range of acceptable error for reproducibility. Thirty percent of the injected pulses are outside of this range, and therefore 70% of the pulses are reproducible with 3.5 hr of continuous sensing. While with 0.1 mL it is possible to detect 20 successive pulses, with 0.4 mL, the error is ±5%. This deviation can be due to a change in the accommodation of the conductive networks produced by the carbon black particles in the polymer matrix. The carbon black particles undergo a modification of their initial or previous pulse. It can be seen that the response and desorption times depend on the THF concentration. For quantities lower than 0.5 mL, the response times tend to be greater than 2–10 s. The sensitivity percent is greater for higher THF quantities, on the order of 180%, and the desorption times tend to be greater than 5 min. Low THF concentrations tend to be retained with higher intensity, and this is evidenced by long desorption times.

It can be seen that the response and desorption times depend on the THF concentration. For concentrations lower than 0.5 mL, the response times tend to be greater than 2–10 s for amounts above 0.5 mL THF. The sensitivity percent is greater than the unit for lower THF quantities, but for higher concentrations than 0.4 mL the sensitivity percent is in the order of 180%, and the desorption time similarly tends to be greater than 5 min. Small THF concentrations tend to be retained with higher intensity, which is evidenced by the long desorption times.

Some authors, such as Kaur et al. [25], note that a short response time in a chemiresistor is 45 s; Chen et al. [26] noted that a sensor of WPU/3.5 wt.% CB exposed to 10 ppt (parts per trillion) of acetone exhibits response times from 150 to 400 s, and a short desorption time for the same sensor ranges from 50 to 100 s. Ding et al. [27] reported a desorption time (less than or equal to 7 s) in a gas sensor based on nanofibers manufactured via electrospinning. There is not a precise time designed to define a short or long response. However, one might conclude that the same compound exhibits a range in the parameters depending on the type of sensor and on the specific application.

4. Conclusions

Conductive layers of modified cellulose and 10 wt.% CB composites were prepared by spinning. Their ability to detect THF vapors was evaluated by determining parameters: sensitivity percent, response and desorption times, reproducibility, and durability in two types of experiments, PST and MST. The sensitivity percent from PST experiments allowed evaluation of the potential to effectively sense THF in the range from 0.1 to 1.0 mL. Sensing effectiveness increased as the amount of THF increased, reaching a maximum of 180% at 1.0 mL THF. The response times for the same experiment were in the range of 2–24 s while desorption times decreased from 12 to 2.3 min. MST experiments were undertaken to determine reproducibility and durability at constant THF volumes, 0.1 and 0.4 mL. For both volumes, the reproducibility was around 65%, and the durability decreased at a half from 20 to 10 pulses when the amount of solvent increases from 0.1 to 0.4 mL. Those results indicate that ACAB with 10 wt.% CB composites effectively detects THF.

Conflict of Interests

The authors of the paper do not have a direct financial relation or conflict of interests with the commercial identities mentioned in this paper. The commercial trademarks, Sigma-Aldrich and Chemical and Electron Microscopy Sciences mentioned in this paper, are only for guaranteeing the reproducibility of the experiments, not leading to a conflict of interests for any of the authors.

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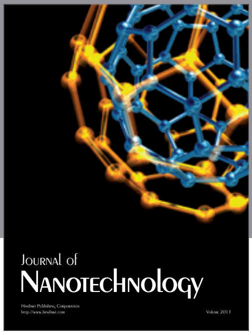
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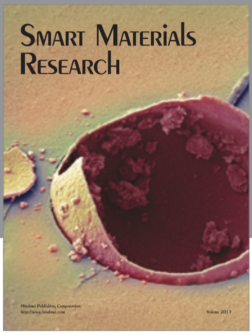
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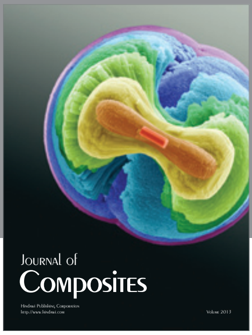
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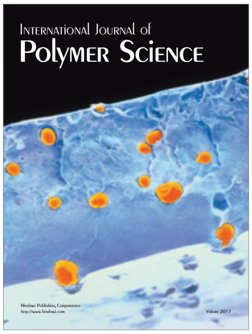
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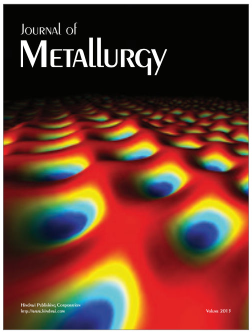
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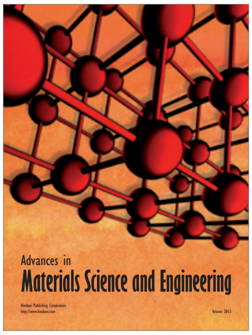
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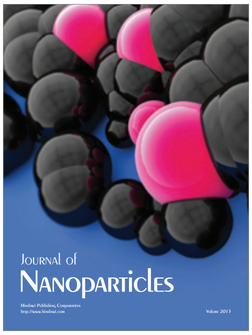
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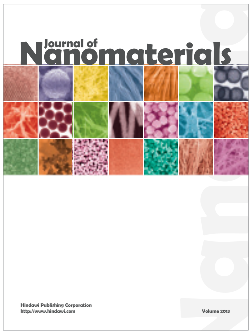
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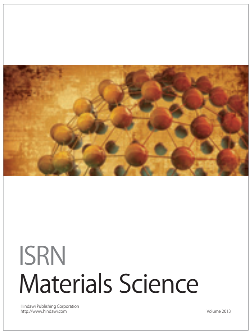
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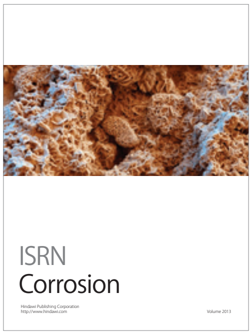
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