

Research Article

Mechanical and Thermal Behaviour of Ecofriendly Composites Reinforced by *Kenaf* and *Caroà* Fibers

P. Persico,¹ D. Acierno,² C. Carfagna,^{1,2} and F. Cimino²

¹*Institute of Chemistry and Technology of Polymers, National Research Council of Italy, Via Campi Flegrei, 34 80078 Pozzuoli (Na), Italy*

²*Department of Materials and Production Engineering, University of Napoli "Federico II", p.le Tecchio, 80 80125 Napoli, Italy*

Correspondence should be addressed to P. Persico, paola.persico@ictp.cnr.it

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Two kinds of environmental friendly composites were prepared based on sustainable matrices, respectively, defatted cross-linked soy flour and thermoplastic polyhydroxybutyrate cohydroxyvalerate, reinforced by natural fibers from *Caroà* and *Kenaf* plants. The obtained composites were compared in terms of moisture tolerance, thermal and mechanical properties, and thermoregulation ability. It was found that this ecofriendly systems have suitable properties for indoor applications in housing and transportation.

1. Introduction

The transition toward a bio-based economy and sustainable developments offers high perspectives for natural fiber markets, and so-called *green* materials are increasingly being explored as substitutes to conventional plastics.

Therefore, plant-based biopolymers are fast becoming a viable alternative to petroleum-based polymers. The benefit of those sustainable resources is that they can be regrown within the foreseeable future, without negative side effects on global biodiversity [1–3].

Among biodegradable polymers, starch, wheat gluten, zein, bacterial polyesters, and soy protein have attracted much attention. The present research deals with the preparation and characterization of composites obtained by reinforcing defatted soy flour (SF) [4–8] and thermoplastic polyhydroxybutyrate cohydroxyvalerate (PHBV) resins [9–12] with two different natural fibers named, respectively, *Caroà* and *Kenaf*.

Neoglaziovia Variegata (Arruda da Camara) Mez is a species which belongs to Bromeliaceae family and is native to the lower stratum of the Brazilian Caatinga. It has striped leaves, flowers protected by bracts with bright coloration, and fruits as juicy berries. This species is known in the

Northeast Region of Brazil as *Caroà* and represents the raw material most used by local craftsmanship. Natural fibers extracted from its leaves are used for manufacturing string, hats, purses, rugs, hammocks, fishing nets, and fabrics [13].

Kenaf, a species of *Hibiscus*, is a short-day, annual herbaceous plant cultivated for the soft bast fiber in its stem. The kenaf stalk is made up of an inner woody core and an outer fibrous bark surrounding the core. The fiber derived from the outer fibrous bark is also known as bast fiber, which has been used traditionally in the manufacture and trade of cordage products due to its superior flexural and tensile strength [14].

It is worth noting that chemical composition and cell structure of natural fibers are quite complicated. In general, they are essentially a composite in which rigid cellulose, consisting of helically wound microfibrils, is embedded in a soft matrix of lignin and hemicellulose [15, 16].

Current innovation on the markets for natural fiber containing products (composites) has widened the scope of their use as, for example, in the building industry that encourages the use of environment-friendly alternative construction materials [17]. For instance, a thermal storage capacity similar with that of conventional building materials can be obtained by the incorporation of microencapsulated

phase change materials (PCMs) in natural composites in order to achieve an effect of thermoregulation [18]. This can contribute to reduce the thermal damping due to external temperature oscillations, making the in-house environment more comfortable.

In this paper, the moisture absorption/desorption capacity, the thermal and mechanical properties, and the thermoregulating ability of prepared ecosustainable composites will be discussed.

2. Experimental

2.1. Materials. Soy flour (defatted SF with 52% protein) was provided by Mangimifici Liverini-Telese (BN-Italy). Kenaf fibers were kindly supplied by Kenaf Eco Fibres Italia S.p.A (Guastalla-Italy) and Caroà fibers were kindly supplied by Federal University of Campina Grande, Brazil.

PHBV ENMAT Y 1000P was supplied by Tianan Biological Material Co., Ltd. Ningbo-China. Analytical grade glycerol (GLY), 99% solution in water and glutaraldehyde (GA), 25% solution in water were obtained from Sigma Aldrich. Microencapsulated PCMs characterized by the phase transition temperature at 28°C (MCPCM28) were supplied by Microtek Laboratories (USA). Microcapsule diameters range from 5 to 40 μm .

2.2. Preparation of Composites

2.2.1. Soy Flour-Based Composites. SF powder was mixed with distilled water in 1:9 ratio (by weight), and, if requested, 15 wt.% of GLY plasticizer was added. The SF suspension was homogenized using a magnetic stirrer for 15 minutes, the pH of the mixture was adjusted to 11 at 70°C, and after 30 minutes 40% by SF weight of GA as cross-linking agent was added, causing colour change of the mixture from olive green to brown, when most of water was evaporated out.

The precured resin was mixed with natural fibers (0.5 cm length) in a Brabender-like apparatus (Rheocord EC of HAAKE, NJ, USA) at room temperature in order to obtain a series of eco-composites. Formulations are listed in Table 1. The blends were dried for 5 days at room temperature and then pressed for 45 minutes at 120°C under 50 bar in a Collin hydraulic hot press for finalizing the curing process.

As for thermoactive soy panels, 10% by SF weight of microcapsules were added to the mixture.

2.2.2. PHBV-Based Composites. Polymer composites containing 40 and 60 wt.% of kenaf fibers were prepared using a lab-scale mixer (Thermo Haake Rheomix) at 190°C and 16 rpm mixing rate for 15 min. Polymer pellets were first fed into the mixer and then fibers (previously dried) were added as soon as the torque indicated melting of the polymer (about 2 minutes), the following 13 minutes being enough to reach torque stabilization that means homogeneous mixing of filler and matrix.

Samples from the mixer were compression moulded at 190°C and 100 bar for 10 min using a Collin polymer

TABLE 1: Eco-composites formulations.

Samples		Kenaf (wt.%)		Caroà (wt.%)	
SOY based	15 wt.% GLY	40	60	40	60
	No GLY	40	60	40	60
PHBV based		40	60	—	

press to form squared plates (thickness 3.5 mm) for further characterizations.

As for thermoactive PHBV panels, 10% by PHBV weight of microcapsules were blended with the matrix.

2.3. Moisture Absorption/Desorption. Moisture uptake of natural fibers and moisture uptake/release of soy-based composites was measured gravimetrically. The samples were conditioned in a vacuum oven at 70°C for 24 h, cooled in a desiccator and immediately weighted.

The weighted samples were then placed in a closed chamber at 90% relative humidity and room temperature for absorption test.

As for kenaf and caroà fibers, the same amount by weight of dried fibers was used for absorption measurements, performed for 60 hours. The water uptake has been calculated by the weight increase of the fibers normalized to their initial weight.

As for absorption test of soy-based composites, the preconditioned and weighted sample (60 mm length, 10 mm width, and 3.5 mm thick) containing 40 wt.% of kenaf was first placed in a closed chamber at 90% relative humidity and room temperature for 25 hours. When the sample reached a constant weight, the desorption measurement was immediately started on the same specimen, putting it in a desiccator filled with silica gel desiccant for 72 hours. The water uptake has been calculated by the weight increase of the specimen normalized to its initial weight.

2.4. Thermogravimetric Analysis. Thermal properties of eco-composites were analysed by TGA. Samples were heated up to 50°C, and 30 min isothermal step was carried on in nitrogen atmosphere, then a ramp from 50°C up to 600°C at a scanning rate of 10°C/min in air was performed by using a TGA Q5000 from TA Instruments.

2.5. Flexural and Impact Tests. Three-point flexural test was performed using an Instron model 4504 machine at a deformation speed of 1 mm/min and with a span length of 48 mm to evaluate the flexural modulus under 1 kN load cell according to ASTM D790 test method.

Fracture tests were carried out with a Charpy Ceast Resil Impactor equipped with a DAS 4000 Acquisition System, using an impact energy of 3.6 J, an impact speed of 1 m/s and a span length of 48 mm. Samples (60 mm length, 10 mm width, and 3.5 mm thick) with a notch depth to width ratio of 0.3 were fractured at room temperature according to ASTM D256 test method. For each material, 5 specimens were fractured, and the average value of resilience was calculated.

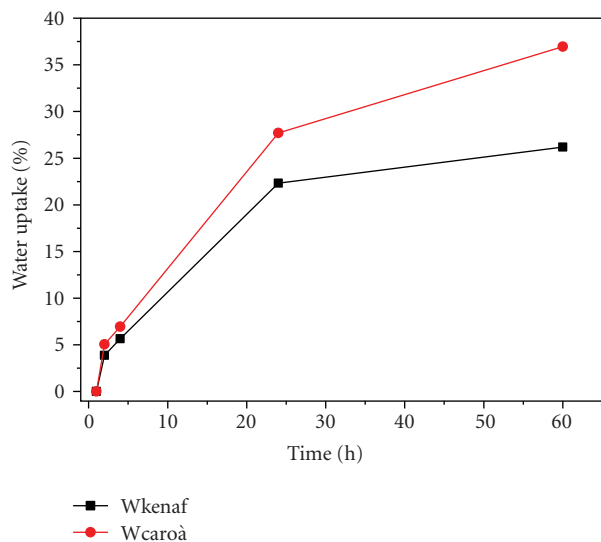


FIGURE 1: Moisture uptake % of caroà and kenaf fibers.

2.6. *Thermoregulating Test.* Time-temperature images during heating and cooling of samples were recorded by means of an infrared thermocamera. The experimental equipment designed to test the thermoregulating effect was composed as follows [19]:

- (1) Peltier-effect device, for heating and cooling the samples,
- (2) plexigas chamber, in order to avoid thermal influences by external environment,
- (3) electrical waves generator, consisting of a PID controller (that checks on the temperature and induces the rely to invert) and the inverter which is an electrical or electromechanical device that produces a sinusoidal waveform,
- (4) infrared thermocamera to record images of thermal performance (FLIR SYSTEMS, Thermo Vision A40 M Researcher).

Specimens size (4 mm length, 2 mm width, 2 mm thick) has been selected in respect of this experimental setup.

3. Results and Discussion

3.1. *Absorption/Desorption Test.* In Figure 1 the percentage of water uptake of caroà and kenaf due to moisture penetration is reported.

As can be noted, caroà absorbs a slight higher quantity of water compared with kenaf. As reported from literature [16], several factors such as density, chemical composition, and structural parameters contribute to the differences in water absorbability of fibers.

The amount of water absorbed is an important factor in composites characterization, since it can have undesired effects on their mechanical properties and dimensional stability, and the hydrophilic character of natural fiber contributes to moisture absorption [20]. Since kenaf is reported

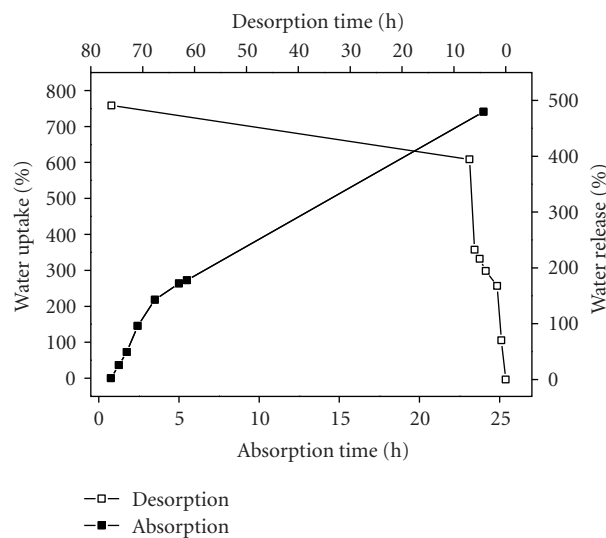


FIGURE 2: Absorption/desorption tests on 40 wt.% soy/kenaf composites.

to have the less moisture content [16] and is found to absorb less quantity of water, only kenaf-based composites were considered for further absorption and desorption tests.

Figure 2 shows the water uptake and release of soy-based composites containing 40 wt.% of kenaf. The weight of the sample increased as a consequence of water absorption up to about 700% of its initial weight in 24 h. The desorption test was started immediately after, placing the same sample in a desiccator filled with the desiccant agent at room temperature. The specimen released the water absorbed in 72 h, without application of any heating process. This “sponge” like behaviour can be considered suitable for indoor applications in wet environment.

It was observed that the moisture penetration does not depend only on the fiber but also on the type of resin in addition to other effect such as the quality of fiber matrix interface, the relaxation of the resin in presence of moisture, the void content in the resin, and the binding of water molecules on its molecular structure [20].

However, adsorption/desorption experiments (not reported) performed on PHBV-based systems revealed that a really small amount of moisture penetration is achieved in thermoplastic composites.

3.2. *Thermogravimetric Analysis.* The thermal resistance of the soy neat resin was compared with that exhibited by the resin reinforced with 40 wt.% of both caroà and kenaf fibers (Figure 3).

Starting from 50°C, when the samples are water-free, they are almost thermally stable in air flux up to 200°C despite of the presence of natural fibers. Probably, their degradation leads to the formation of some ash that can hinder further oxygen diffusion through the resin.

TGA traces of PHBV and its composites are shown in Figure 4. The curves revealed that PHBV composites undergo massive thermal degradation above 250°C. The

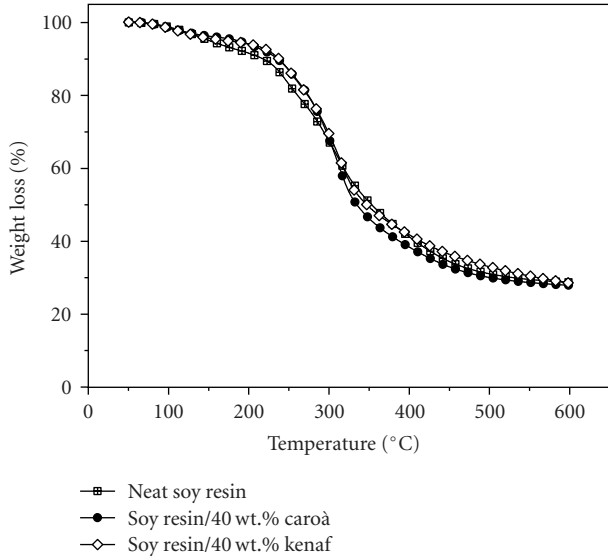


FIGURE 3: Thermogravimetric curves for soy-based systems.

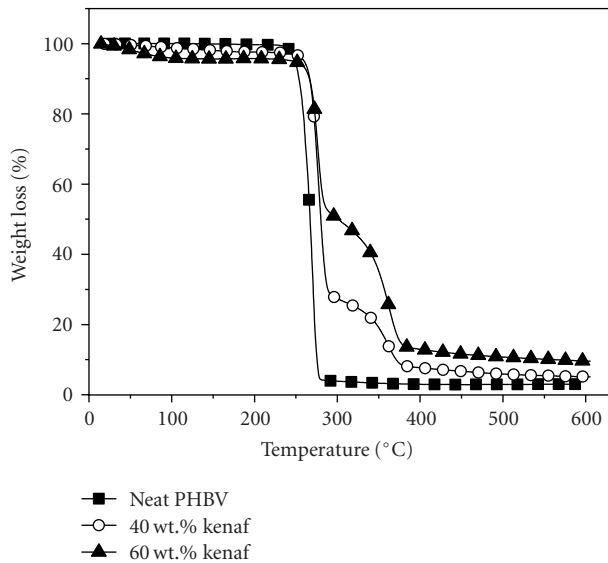


FIGURE 4: Thermogravimetric curves for PHBV-based systems.

onset of thermal degradation of composites was similar to that of neat PHBV. This can be attributed to the fact that PHBV is thermally unstable and starts degrading drastically above 250°C due to chain scission reactions leading to the reduction of molecular weight and resulting in the formation of the volatile acid products such as crotonic acid. Crotonic acid is also supposedly known to cause hydrolysis of the cellulose content of natural fiber and could result in defibrillation [21]. TGA curves of composite systems also suggested that there was no additional thermal degradation of PHBV with incorporation of kenaf fibers. There was formation of different amounts of residue, the amount was higher in composites having a higher content of fibers.

3.3. Mechanical and Impact Properties. Mechanical tests were performed in order to evaluate the flexural ultimate

TABLE 2: Flexural ultimate strength of soy composites.

Kenaf (wt.%)	Flexural strength (MPa)	
	0 wt.% gly	15 wt.% gly
40	24.5 ± 5.8	7.0 ± 0.4
60	29.5 ± 1.2	13.7 ± 3.4
Caroà (wt.%)		
40	27.4 ± 6.1	9.6 ± 0.4
60	30.3 ± 7.1	21.4 ± 4.6

TABLE 3: Flexural and impact properties of PHBV composites.

Samples	Flexural modulus (MPa)	Flexural strength (MPa)	Impact strength (KJ/m ²)
Neat PHBV	1280 ± 120	28.2 ± 1.1	0.85 ± 0.15
40 wt.% kef	3605 ± 230	16.7 ± 4.0	2.25 ± 0.32
60 wt.% kef	4020 ± 370	14.1 ± 3.5	1.70 ± 0.10

strength of soy based composites. The results are listed in Table 2. Both kenaf and caroà systems not containing glycerol displayed quite similar values of flexural strength. When glycerol is used in the formulation flexural values are lower, confirming its plasticization efficiency for the soy resin [22, 23]. Additionally, the higher is the fibers content, the higher is the flexural strength caused by the bridging formed in between them.

Flexural and impact properties of kenaf reinforced thermoplastic composites are listed in Table 3. The flexural modulus is representative of intrinsic plastic deformation ability of virgin matrices, and PHBV exhibits a very limited deformation to fracture in comparison with other thermoplastic polymers. Nevertheless, PHBV composites show a significant increase of flexural modulus values with respect to the corresponding neat matrix due to the presence of fiber reinforcement [24]. This finding could suggest that there is a considerable interfacial interaction in between the fibers and PHBV matrix, and, at the same time, fibers entanglements act as resistant junction points against deflection forces.

It should be reminded that in a flexural test the combination of tensile and compressive strength occurs, and therefore, any measurement is affected by both flexural and shear stresses [25]. It is interesting to analyze the flexural strength values of thermoset and thermoplastic composites. In the case of the soy-based system not containing glycerol, the flexural strength remains constant independently of fibers content. On the other hand, by using the glycerol plasticizer, it is possible to reduce the soy system brittleness, and this effect is more pronounced when 60 wt.% of fibers is used.

As for PHBV, the response of the composite based on a brittle matrix will be affected by the properties of the constituents and by the interaction between them. It was observed that flexural strength decreased as a result of the incorporation of stiffness and flexible fibers.

The impact strength of a short fibre composite is determined by the energy required for the plastic deformation of matrix and fibers, for the fracture of matrix and fibers, for

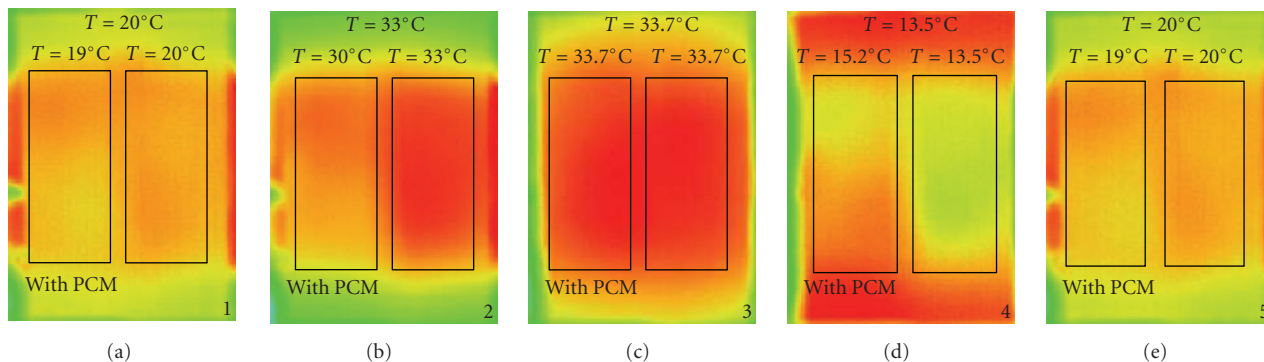


FIGURE 5: Colour images of soy resin measured by infrared thermocamera.

fiber-matrix debonding at the interface and for overcoming the friction following the debonding during pullout [26]. The nature of composite, fibers, and type of impact test are critical factors for the increase or decrease in the apparent impact strength. An Izod impact test is basically a flexural test with a transient load being applied. In the case of composites, their characteristics are related to the ability of both the matrix and the fiber-matrix interface to absorb the high speed impact.

Low fracture toughness was measured for neat PHBV, but the addition of fibers enhances its impact performances. The slight improvement from 0.85 up to 2.25 kJ/m² can be attributed to the energy dissipation mechanism occurring as a consequence of the good level of fiber-matrix adhesion and fiber aspect ratio (l/d).

3.4. Effects of Thermoregulation. Infrared (IR) thermographic systems provide images that represent surface temperatures by measuring the magnitude of infrared radiation emitted by the surface of an object. To perform the temperature distribution analysis, a thermal vision camera can be used to detect IR radiation and convert this information into an image, where each pixel corresponds to a temperature value. Modern IR imagers resolve surface temperature differences of 0.1°C or less. With this high sensitivity, they can provide a nondestructive evaluation of thermal phenomena, which are only revealed in the form of slight temperature gradients. Infrared thermography can be used as both a qualitative and a quantitative tool. The thermal qualitative effect due to the presence of micro-PCMs can be evaluated by recording colour images using the infrared thermocamera, as displayed in Figure 5.

The soy composite sample containing PCMs (reported on SX side of each frame) placed on the surface of the Peltier cell is able to delay and soften the thermal response in comparison with the sample without PCMs (reported on DX side of each frame), since microcapsules absorb heat during the melting process of paraffin waxes [19].

Temperature magnitude in a thermograph can be visualized by a colour scale in which each colour is related to a temperature range. From the hottest to the coldest temperature, the colours are so scaled: red, orange, yellow, green, and blue, the exact value depending on the experimental set fixed. In

this experiment, the image was equivalent in the temperature range from 19°C to 40°C.

When the heating/cooling cycle starts, the samples are at the same temperature (step 1), both showing yellow colour ($T = 19^\circ\text{C}$). During the heating cycle, the sample containing PCMs (SX side) turns orange, while reference composite (DX side) is red (step 2). Such colour differences indicate that the temperature of the sample containing PCMs is lower than that of composite not containing them. In this process, PCMs act as a thermal buffer material by absorbing heat delaying the heating of the panel.

When the heating cycle stops, the samples are at the same temperature as confirmed by the red colour for both (step 3, $T = 33.7^\circ\text{C}$).

During the cooling cycle, the sample with PCMs is orange, while reference composite is green (step 4). This colour difference indicates that the temperature of the sample with PCMs is higher than that of reference composite, since part of the available heat is used for wax crystallization, postponing the panel cooling. When the cooling cycle stops, samples reach the same temperature and their colour is orange (step 5).

The same qualitative behaviour (not reported) was observed on PHBV thermoactive eco-composites: the thermoregulation features are related to the presence of microencapsulated PCMs despite the substrate considered. Moreover, it is possible to tailor the thermoregulation efficiency of panels by using more or less amount of microcapsules in the formulation. In general, the higher is the PCMs content, the greater is the thermal buffer extent.

4. Conclusions

In this work some characteristics of SF- and PHBV-based composites reinforced by kenaf and carob fibers have been studied in order to evaluate their potential in technological terms. These composites based on natural fibers and eco-sustainable matrices, both thermoset and thermoplastic, are expected to represent a new generation of materials engineered for secondary applications in the field of buildings construction or for the design of innovative bioarchitecture solutions.

As for the moisture uptake/release, soy/kenaf systems act in the same way as a sponge: they are able to take water and then lose it in a dry surrounding environment without forced heating procedures. On the contrary, PHBV/kenaf composites are not hygroscopic, thus representing a promising starting point for durability.

The mechanical characteristics of kenaf reinforced PHBV panels obtained by hot pressing suggested that fibers are able to improve the flexural modulus while promoting the dissipation of impact energy. The soy/kenaf composites mechanical properties are affected, on the other hand, by the use of glycerol as resin plasticizer.

The thermoregulation ability of all the systems can be tailored by using more or less amount of microcapsules in the formulation. In general, it was observed that the higher is the PCMs content, the greater is the thermal buffer extent. However, the thermal stability of thermoset and thermoplastic composites was found to be approximately the same up to 250°C.

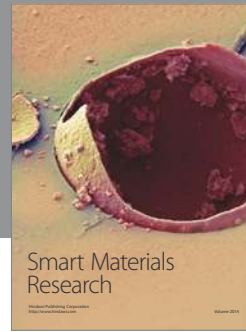
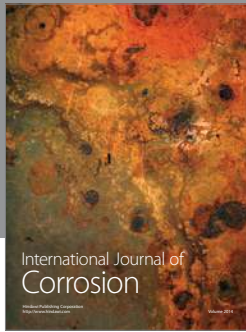
Acknowledgments

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