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RESEARCH ARTICLE

Influence of Magnesium Oxide Nanoparticles on the Compressive Strength of Urea Formaldehyde Resin

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Abstract

Urea Formaldehyde (UF) resins have good chemical resistivity and high thermal stability, making them an excellent choice in the construction industry. They, however, pulverize quickly and have low strength and toughness. In this work, magnesium oxide (MgO) nanoparticles were added to UF as nanofillers to influence its compressive strength. MgO nanoparticles were synthesized by reducing magnesium nitrate at different concentration, using orange peel extract. X-ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) techniques were used to confirm the formation of MgO nanoparticles. XRD results showed the formation of 43 nm, 35.28 nm, and 32.5 nm sized nanoparticles for 0.1 M, 0.2 M, and 0.4 M concentrations respectively. The varying-sized MgO nanoparticles were used for the preparation of UF/MgO nanocomposite at different weight-percentage (wt-%) ratios. The comparative study on the compressive strength of Urea Formaldehyde resins and UF/MgO was performed. From the results it was found that the addition of MgO nanoparticles to UF resin enhances the compressive strength at certain wt-% ratios.

Keywords: Magnesium oxide, XRD, FTIR, urea-formaldehyde, nanocomposites

Introduction

Nanostructured crystalline particles have caught the interest of researchers attributable to the wide range of applications made possible by their particle size-dependent properties, as well as their scientific and industrial significance [1]. Nano-sized particles of metal oxide materials have received attention in various industrial, medical, environmental, and agricultural applications [2]. Recently, metal oxide nanoparticles are also used as antibacterial agent [3]. These nanomaterials have distinct thermal, structural, and electronic properties that entrust them with a high level of scientific interest in both basic and applied fields [4]. One metal oxide, i.e., magnesium oxide (MgO), has high thermodynamic stability, low dielectric constant, and large band gap, making it a material of magnificent technological importance in the construction industry [5]. MgO has non-combustible properties, high stability, a high specific surface area, and a higher specific heat capacity, making it effective toughening filler without compromising the properties on which they are added. Researchers are currently investigating materials with the properties of high thermal stability, good chemical resistivity and excellent flame retardancy for the use in construction materials to increase the safety.

UF has been promoted as a suitable choice for satisfying the aforementioned construction industry demands[6].This increases the wide application range of UF composite. However, UF lacks active functional groups due to which it is brittle, pulverizes quickly, and

has low strength and toughness[7]. For these reasons, it is crucial to focus on improving the mechanical properties of UF, such as compressive and bending strength, friability, and pulverization rate. Thus, to improve the mechanical strength of UF, research has recently emphasized incorporating reinforcing agents. The toughness of UF can be improved by two methods: physical and chemical. Flexible groups are integrated into the macromolecular chains of polymers using the chemical technique, which impacts UF's strong performance due to the inclusion of other polymers [8]. External toughening agents are directly absorbed into UF by producing a mixture in the physical approach, which is believed to be a better method to improve the mechanical properties of UF resin [9]. Toughening agents are classified as organic or inorganic. In order to improve mechanical properties, organic toughening agents such as polyvinyl alcohol (PVA) [10] polyethylene glycol (PEG) [11] fiber[12], cellulose [13] and inorganic toughening agents such as MgO nanoparticles [14], silica gel [15], zirconium [16] ,are widely used. Inorganic nanoparticles have larger specific surface areas, more defects, and more surface atoms than organic toughening agents, which could combine closely with UF and improve its ability to bear a load. Furthermore, nanoparticles can effectively pass external stress and absorb a large amount of energy when subjected to external force [17]. As a result, inorganic nanoparticles have received a lot of attention to improve the toughness of polymers. However, no studies have been conducted on the effect of MgO nanoparticles on the compressive strength of UF resin. It would be good constructive materials with improvised the brittle properties of UF resin with nanoparticles. Therefore, this study focuses on the synthesis of MgO NPs from green route and using the synthesized NPs for developing a UF/MgO nanocomposite. Furthermore, this work focuses on the investigation of the compressive strength of UF/MgO nanocomposite prepared at different wt-% ratios and comparing it with the compressive strength of pure UF resin.

Materials and Methods

Materials

Citrus sinensis (sweet orange) peels were collected from fruit shops in Kathmandu. The chemicals used in the experiments were magnesium nitrate $Mg(NO_3)_2$, sodium hydroxide NaOH, urea, and formaldehyde manufactured by Merck-India Pvt. Ltd. and obtained from a local supplier in Kathmandu. All of the chemicals were of analytical grade and were used without further purification.

Methods

Preparation of Extract from Orange Peels

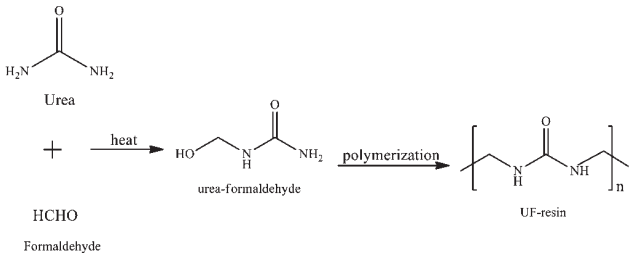
Dried orange peels were ground into powder and 40 gm of the powder was mixed with 400 mL of deionised water in an R.B flask; the mixture was then refluxed for 1 hour. The extract was filtered through Whatman filter paper no. 41 [18].

Preparation of MgO Nanoparticles

Three different magnesium nitrate $Mg(NO_3)_2$ solution was used as an initial precursor having 0.1 M, 0.2 M, and 0.4 M concentrations. Peel extract was added into various magnesium nitrate solutions which were stirred for 4 hours continuously by using magnetic stirrer. The pH of the solution was maintained 12 by adding NaOH solution drop wisely. The particles formation occurs during stirring process where magnesium nitrate was reduced to magnesium oxide [18].

Preparation of Urea Formaldehyde (UF) Resin and UF/MgOnanocomposite

UF was prepared by combining its precursors, urea and formaldehyde, in a 1:2 ratio. The reaction mixture was stirred in a water bath at 60 - 70 °C. The 20 % NaOH solution was added drop wise to maintain the basic conditions, *i.e.,* pH 10. The prepared UF resin was poured into a 2 cm cubical wooden mould. The UF/MgOnanocomposites with various weight-percentage ratios of MgO (*i.e.,* 1 %, 2 %, 3 %, 5 %, and 10 %) were prepared following same procedure as mentioned above. Finally, the UF resin was pre-cured in an oven at 60 °C for 12 hours. After that, it was post-cured for 12 hours at 120 °C[19].The reaction of urea and formaldehyde is shown in scheme 1.



Scheme 1. The polymerization reaction of UF-resin

Characterization technique

X-ray diffraction (XRD) and Fourier- Transform Infrared (FTIR) analysis

XRD technique was used to calculate the crystalline size and structure of the synthesized MgO nanoparticles. The crystal phase and structure of the prepared samples were determined by using Bruker D2 PhaserDiffractometer (USA), with a monochromatic CuKα radiation source (λ= 0.15418 nm) at angle 2θ ranging from 10° - 80°.

The FTIR spectroscopic technique analysed the vibration frequency of the molecules' stretching and bending modes of synthesized MgO NPs and UF resin. The formation of synthesized MgO NPs and UF resin was confirmed using the instrument IR Affinity-1S FTIR Spectrometer (SHIMADZU, Japan), where spectra were analysed using the KBr pellet method in the spectral range of 4000 – 400 cm^{-1}.

Compressibility Test

The compressive strength test was performed to determine the maximum compressive load that a material can withstand before breaking. The following equation is used to calculate the compressive strength of synthesized MgO/UF nanocomposites[20]. The equation used for the calculation is

$$\text{Compressive strength} = \frac{\text{(Breaking load)}}{\text{Cross–sectional area}} \quad \ldots\ldots (1)$$

The compressive strength of the prepared sample block was tested by using Compression Testing Machine (C.T.M.), Harrish and Terrish, India, having factor 0.14.

Results and Discussion

XRD Analysis of MgO Nanoparticles

The crystalline phase and structure of synthesized MgO nanoparticles from sweet orange were determined using the XRD analysis technique, where the crystalline size was calculated using the Debye-Scherrer equation[21]. The XRD pattern of MgO nanoparticles produced with 0.1 M, 0.2 M, and 0.4 M magnesium nitrate and 0.2 M sodium hydroxideis shown in Figure 1.

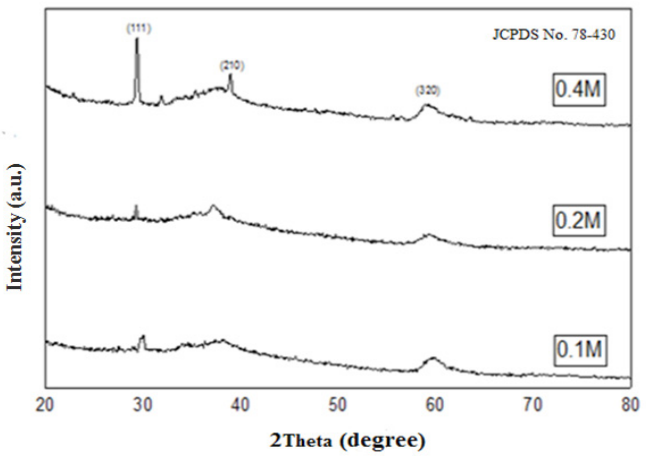


Figure 1. XRD pattern of MgO NPs synthesized using 0.1 M, 0.2 M, and 0.4 M $Mg(NO_3)_2$

The diffraction pattern (Figure 1) shows various peaks, corresponding to (111), (200), and (220) refection planes assigned to an angle 30°, 40°, and 60° respectively as per the JCPDS No. 78-430representing the cubic structure of MgO[22]. The average crystallite size of synthesized MgO nanoparticles from 0.1 M, 0.2 M, and 0.4 M $Mg(NO_3)_2$ was 43 nm, 35.28 nm, and 32.5 nm respectively.

Fourier Transform Infrared (FTIR) analysis of synthesized MgO nanoparticles and Urea-Formaldehyde (UF) resin

The FTIR spectroscopic analysis was done to analyse the functional group present in the synthesized samples. Figure 2 represents the FTIR spectra of synthesized MgO nanoparticles with 0.1 M, 0.2 M, and 0.4 M $Mg(NO_3)_2$.

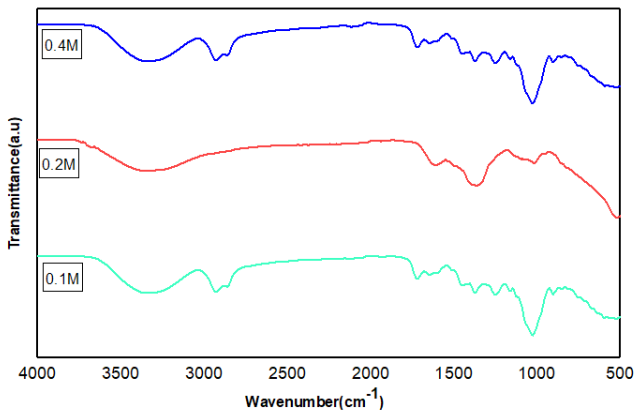


Figure 2. FTIR spectra of MgO NPs synthesized using 0.1 M, 0.2 M, and 0.4 M $Mg(NO_3)_2$

The spectra of the synthesized MgO nanoparticles are illustrated in Figure 2. The peaks at 3340.71cm^{-1}, represented the stretching vibration of the O-H group due to water molecules present in the precursor solution. The peak at 1643.35cm^{-1} corresponds to the bending vibration of a surface hydroxyl group (-OH) [23].The peaks at 1373.32cm^{-1} and 1026.13cm^{-1} are assigned to O-C=O bending and bending vibration of water molecules[24]. The vibrations at 2924.09cm^{-1}, and 2862.36cm^{-1} are due to C-H bond.The peak observed at 524.64cm^{-1} indicates the formation of MgO nanoparticles[25][26].

Moreover, the FTIR spectrum of pure urea-formaldehyde (UF) is shown in Figure 3.

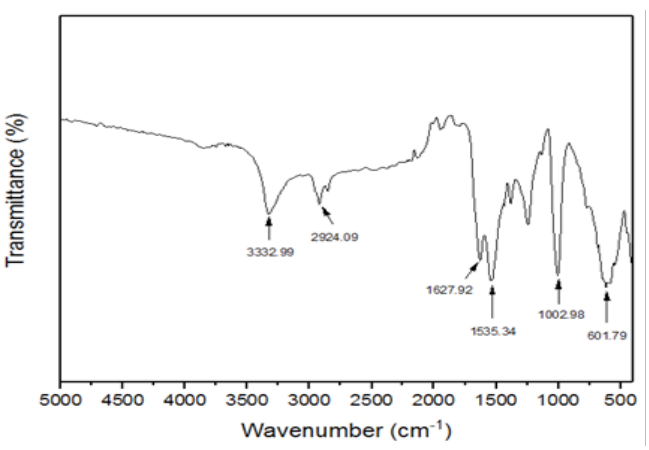


Figure 3. FTIR Spectrum of Urea-formaldehyde (UF) resin

The spectrum of prepared UF resin shows the peak at 3332.99cm^{-1}, 2924.09 cm^{-1}, 1627.92cm^{-1}, 1535.34cm^{-1}, 1002.98cm^{-1} and 601.79cm^{-1}. The peak at 3332.99cm^{-1} corresponded to N-H stretching of primary aliphatic amines, and the peak at 2924.09cm^{-1} represented C-H stretching of UF. The peak at 1627.92cm^{-1} and 1535.34cm^{-1} attributed to the presence of –NH-CO-NH- and –CO-NH- groups. The peak at 1002.98cm^{-1} corresponded at the $–CH_2OH$ group [27]. The FTIR spectrum confirmed the formation of urea-formaldehyde resin using precursors, urea, and formaldehyde.

Compressive Strength Analysis of UF/MgONanocomposites

The compressive strength test of pure UF resin and UF/MgO nanocomposites prepared by varying the wt-% (1 %, 2 %, 3 %, 5 %, and 10 %) ratios of MgO was investigated by using the equation (1) at room temperature of 25 °C.

The prepared UF and UF/MgO nanocompositeswere compressed between the platens of the Universal Compressive Strength Testing Machine with a factor of 0.14 by a gradually applied load. The obtained data are presented in Table 1.

Table1. Compressive Strength of UF/MgOnanocompositesat different composition

S.N.	MgO NPs (wt.-%)	Breaking Load(N) in concentrations			Compressive Strength (MPa) in concentrations		
		0.1 M	0.2 M	0.4 M	0.1 M	0.2 M	0.4M
1	1%	280	285	292	70	71.25	73
2	2%	289	295	300	72.25	73.75	75
3	3%	305	311	315	76.25	77.75	78.85
4	5%	325	340	340	81.25	85	85
5	10%	282	285	290	70.5	71.25	72.5

The compressive strength of pure UF resin was calculated to be 28 MPa. The results of our work showed that UF/MgO nanocomposites have higher compressive strength than pure UF resin. The compressive strength of the UF/MgO nanocomposites increased as the wt-% ratio of MgO nanoparticles increased. It means that the addition of MgO nanoparticles to UF resin resulted in a significant improvement of compressive strength. This is due to dispersion of MgO nanoparticles.

The highest compressive strength of 85 MPa was recorded in a nanocomposite containing 5 % MgO in

both 0.2 M and 0.4 concentrations. The compressive strength of nanocomposites increased up to 5 % (wt- %) of MgO nanoparticles, while compressive strength decreased beyond that. This may be due to the effect of agglomeration resulting from a higher percentage of MgO nanoparticles on the cross-linking of UF resin. The compressive strength of the UF resin was found to be increased with the addition of MgO nanoparticles. This explains the use of MgO nanoparticles to improve the strength of materials like UF resin, which is widely used in manufacturing plastics, adhesives, hinges, etc. Similar results were recorded in the previous article when nanoparticles were incorporated in resins [16][28][29].

4. Conclusion

Magnesium oxide (MgO) nanoparticles were synthesized using sweet orange peel extract and different concentrations of magnesium nitrate solution, *i.e.,* 0.1 M, 0.2 M, and 0.4 M. The synthesized MgO nanoparticles were characterized using XRD and FTIR spectroscopic analysis techniques. The XRD pattern showed that the synthesized MgO nanoparticles prepared from 0.1 M, 0.2 M, and 0.4 M $Mg(NO_3)_2$ were cubic structures with an average crystallite size of 43 nm, 35.28 nm, and 32.5 nm, respectively. As the concentration of precursor was increased, the average crystallite size was found to be slightly decreasing. Likewise, UF resin was prepared by using pure urea and formaldehyde resin. The FTIR spectra confirmed the formation of MgO nanoparticles and UF resin. Furthermore, UF/MgO nanocomposites were synthesized by varying the MgO weight percentage of nanoparticles (1 %, 2 %, 3 %, 5 %, and 10 %), and their compressive strength was investigated. It was found that adding MgO nanoparticles to the UF resin increases its compressive strength as the compressive strength of a nanocomposite containing MgO (5 wt.-%) nanoparticles was found to be the highest at 85 MPa. However, the additional increment of MgO (10 wt.-%) nanoparticles showed lower the compressive strength in all UF/MgO nanocomposite.

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