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Preparation and characterization of melamine formaldehyde organo clay nanocomposite foams (MFCNCF)

ABSTRACT

Melamine formaldehyde resins are one of the well-known thermosetting resins. It has been observed that melamine formaldehyde foam composites prepared with various nanoparticle reinforcements exhibit better mechanical, thermal and sound insulation properties. In this study, it was aimed to synthesize melamine formaldehyde organoclay nanocomposite foams and investigate their thermal insulation and mechanical stability by using microwave irradiation and heating technique together, which can offer advantages such as faster reaction, high yield and purity, reduced curing time. Pure melamine formaldehyde foam, MFF, and melamine formaldehyde organoclay nanocomposite foams, MFCNCFs, prepared with various organoclay contents were characterized by HRTEM, FTIR, SEM and XRD techniques. From spectroscopic and microscopic analyses, it was observed that organoclay platelets could be exfoliated with increasing clay content without undergoing much change in the resin matrix. The highest compressive strength was obtained in the MFCNCF3 foam with high organo clay content (0.68 N/mm²) and the bulk density was determined to be quite low (0.20 g/cm³). On the other hand, in the nanocomposite with 0.15 % organo clay content (MFCNCF2), a compressive strength of 0.32 N/mm² and a thermal conductivity coefficient of 0.065 W/m·K were measured.

Keywords: Melamine formaldehyde, Melamine formaldehyde organo clay nanocomposite, foam, Thermal insulation, Compressive strength

INTRODUCTION

Nanotechnology, which is used to refer to the design, construction and use of functional structures with at least one characteristic dimension measured in nanometers, has become increasingly popular in recent years.^{1,2} Nanoparticles, which form the basis of nanotechnology, are incredibly small particles with a size of less than 100 nm and can contain carbon, metal, metal oxides or organic substances.³ One of the most important features of nanotechnological studies is that the basic physical and chemical properties of a material or system at the nanoscale (e.g., melting temperature, thermal conductivity, load capacity, electronic conductivity, tensile strength and color) can be designed as desired and used in many different areas.⁴ Nanoparticles provide improvements in the functionality of metal, ceramic, polymer or composite systems and are used in the development and production of many products in daily life.⁵ Nanoparticles appear to have different physical, chemical, and biological properties compared to their larger counterparts, contributing to effects such as increased chemical reactivity, stability, or greater surface area relative to volume, greater mechanical strength, etc. Among the diverse uses of nanoparticles is polymer clay nanocomposites.⁶

Polymer clay nanocomposites can be made by directly mixing two aqueous solutions containing monomer and clay suspension, respectively, and subsequent polymerization induced by the addition of thermal or light sources or chemical oxidants.⁷⁻¹² Layered silicates are among the most widely studied nanofillers and are used in modified form. It is known that organoclay or silicates generally improve the properties of nanocomposites compared to traditional macro and micromaterials.¹³⁻¹⁵ Organoclay particles are dispersed in the polymeric matrix in three different ways: phase separated (micro-composite) structure, intercalated (intercalated layered) structure and exfoliated (dispersed) structure.¹⁶⁻¹⁸ There is also a mixed structure in which exfoliated and intercalated structures are seen together.

Three main techniques are used to obtain polymer/clay nanocomposites. These techniques differ according to the starting material and the preparation method. They are in situ (simultaneous) polymerization method, solution intercalation method and melt intercalation method (simultaneous) polymerization.^{7,19,20} In situ polymerization method is a particularly suitable method for the preparation of thermoset polymer clay nanocomposites, but it has also been used for thermoplastics.²¹⁻²⁹

Melamine formaldehyde (MF) is a hard, very durable and versatile resin with high flame and temperature resistance, and is synthesized by condensation of melamine and formaldehyde. MF resins have good flame-retardant properties since they release nitrogen gas during combustion. It is emphasized in the literature that the mechanical, thermal and barrier properties of MF nanocomposites are not at the desired level. Improving these properties with various methods is of great importance in terms of the field of use of nanocomposites. It can be said that there is a significant gap in the literature on this subject. For this reason, the aim of the presented study was to synthesize nanoclay reinforced, cross-linked melamine formaldehyde organo clay nanocomposite foams (MFCNCF) by microwave irradiation technique and to investigate the thermal, mechanical and morphological properties of these composites.

MATERIALS and MERHOD

Raw montmorillonite (MMT), supplied from Karakayalar A.Ş. in Çankırı province, Türkiye, with a specific surface area of 64.2 m^2/g and X-ray Fluorescence (XRF) composition given in Table 1, was used for the synthesis of organo montmorillonite (OMMT) to be used in the preparation of nanocomposites.

 Table 1. X-ray Fluorescence (XRF) chemical compositions of Montmorillonite (MMT).

Component (%)		
SiO ₂	59.32	
Al ₂ O ₃	17.19	
Fe ₂ O ₃	5.95	
MgO	3.63	
CaO	2.21	
Na ₂ O	1.68	
K ₂ O	0.97	
TiO ₂	0.74	
SO₃	0.51	
Other	7.81	

Melamine, formaldehyde (37 wt%), NaOH, acetic acid, Tween 80, a nonionic surfactant (polyoxyethylene (20) sorbitan monooleate), and analytical grade glycerin (all supplied by Merck KGaA, Germany) were used for the preparation of melamine formaldehyde (MFF) and melamine formaldehyde organoclay composite foams (MFCNCF). In addition, gasoline, a mixture of isooctane, butane, and 3-ethyltoluene used for foaming, was supplied by a local gas station.

MERHOD

Preparation of organo clay

Montmorillonite (MMT), a cationic surfactant, cetyltrimethylammonium bromide and CTAB (Merck Co.) suspensions, a hydrocarbon material that is a product of petroleum refining and some of the properties of which are given in Table 2, were used to prepare organoclay by solution intercalation method. The procedure applied for the synthesis of organo-montmorillonite (OMMT) is the same as in our previous work.^{30,31}

Table 2. Some characteristics of the hydrocarbon material.

Density (15°C), kg/m ³	990.7
Calorific Value MJ/kg	42.74
Flash Point °C	105.8
Water by Distillation, wt. %	0.1
С	83.4
Н	11.9
N	0.8
S	1.5
Ash	0.03

Preparation of melamine formaldehyde foam (MFF) and melamine formaldehyde organo clay nanocomposite foams (MFCNCF)

Melamine and formaldehyde (37% by weight) (mass ratio: 0.67:0.33) were placed in a three-necked flat-bottomed flask with a thermometer and cooling equipment connected, then mixed with a magnetic stirrer and then heated under reflux until dissolution at approximately 60°C. The pH was adjusted to 8.5 with 40% by weight NaOH solution. Considering the viscosity of the mixture, it was heated under reflux at approximately 95°C for 60 minutes. Then, a certain amount of concentrated acetic acid, 1.0 wt.% glycerin and Tween 80 as well as 6.0 wt.% gasoline was added to the obtained prepolymer and mechanically mixed vigorously to homogenize the mixture. In addition, when preparing untreated melamine formaldehyde foam (MFF), the same processes were performed using only melamine and formaldehyde (37% by weight) (mass ratio: 0.68:0.32). When preparing the MF-organoclay nanocomposite via in situ synthesis, 0.1%, 0.15% and 0.45% organoclay was added to the container by weight. For foam synthesis, the mixture was exposed to microwave radiation in a microwave oven for 2 minutes in a suitable container. Finally, the mixture was placed in a modular square aluminum mold with a volume of 10.0 x 10.0 x 1.0 cm³ and cured by heat treatment at 140°C for 1 hour in a hot air heated oven to remove water and residual formaldehyde and complete the process.

Table 3. Sample Codes and Contents of Pure MelamineFormaldehyde Foam (MFF) and MF-organoclay NanocompositeFoams (MFCNCF1-3).

Sample Codes	Nano Filler	(%w)
MFF	-	-
MFCNCF1	Organo clay	0.10
MFCNCF2	Organo clay	0.15
MFCNCF3	Organo clay	0.45

Spectroscopic and microscopic analyses and measurements of compressive strength and thermal conductivity

Structural, crystallographic and textural characterization of virgin MF foam and MF organo clay nanocomposite foams were performed using spectroscopic techniques such as XRD and FTIR and microscopic techniques such as HRTEM and SEM.

XRD diffractograms for the prepared samples were obtained using a PANalytical Empyrean X-ray diffractometer with Cu Ka (1.540 Å) radiation operating at 5 kV and 40 mA for 2h in the 9°– 90° range and a scan rate of 4/min (Malvern PANalytical Ltd., United Kingdom).

FTIR spectra were obtained using a Vertex 70V FTIR spectrometer with a mid-IR ceramic source in the range of 4,000 to 400 cm⁻¹, an average of 100 scans and a resolution of 1 cm⁻¹ (Bruker Optics Inc., USA).

HRTEM images of the samples were taken using a HITACHI HT7700 high-resolution transmission electron microscope (LaB6 filament) operating at 120.0 kV (Hitachi Ltd, Japan).

SEM patterns of virgin MFF and MF organo clay nanocomposite foams were taken using SEM (FEI-INSPECT S50 model) at 30 kV.

Compressive strength of virgin MFF and MFCNCF foams were performed using a universal testing machine (Zwick/Roell) according to DIN ISO 844:2009-10 standard. The thermal conductivity coefficients of virgin MFF and MFCNCF foams were also measured using a thermal conductivity meter (Quick Thermal Conductivity Meter QTM-500, Japan) with a probe consisting of a single heating wire and a thermocouple.

RESULTS

Textural characterization of melamine formaldehyde foam (MFF) and melamine formaldehyde organoclay nanocomposites (MFCNCF)

In order to compare the differences in the textural structures of raw montmorillonite (MMT) and organo-montmorillonite (OMMT) and to see the effectiveness of the modification, HRTEM images of both were taken and are given in Figure 1. The dark long-fiber lines seen in the HRTEM images indicate clay layers with their 2:1 layered structure. HR-TEM images of melamine formaldehyde foam (MFF) (a) and melamine formaldehyde-organoclay foams containing varying organoclay ratios (MFCNCF1-3 (b-d)) are given in Figure 2.

Figure 2a shows that pure melamine formaldehyde resin is formed as agglomerated microspheres and the aggregates develop in three dimensions.^{32,33} This textural arrangement is due to the use of microwave irradiation-assisted foaming method before curing. Figure 2b shows that due to the exfoliation of organoclay layers into the polymer matrix, MF molecule clusters are regularly sparse or separated while maintaining their spherical structures.³⁴ As the amount of clay increases, the structures of spherical MF molecules are thoroughly separated (Figures 2c and 2d).

Surface morphological characterization of melamine formaldehyde foam (MFF) and melamine formaldehyde organoclay nanocomposite foams (MFCNCF)

SEM patterns taken for pure melamine formaldehyde foam (MFF) and MF-organoclay nanocomposite foams (MFCNCF1-3) are given in Figure 3.

It can be seen that Figure 3a clearly reflects the surface morphology of pure melamine formaldehyde foam (MF) formation. This textural structuring shows that the microsphere clusters develop as three-dimensional branched structures.^{32,35,36} However, it can be seen from Figure 3b that the branched structure almost disappears and the MF molecule clusters show regular stacking, resulting in a more uniform surface morphology due to the exfoliation of organoclay flakes in the polymeric matrix. It can be seen from Figures 3c and 3d that the morphological structures are similar.



Figure 1. HRTEM images of raw montmorillonite (MMT) (a), Organo-montmorillonite (OMMT) (b).



Figure 2. HR-TEM images of melamine formaldehyde foam (MFF) (a) and melamine formaldehyde-organoclay foams containing varying organoclay ratios (MFCNCF1-3 (b-d).



Figure 3. SEM patterns of pure melamine formaldehyde foam (MFF) (a) and MF-organoclay nanocomposite foams containing different clay ratios (MFCNCF1-3) (b-d).

Analysis of FT-IR spectra of melamine formaldehyde foam (MFF) and melamine formaldehyde organoclay nanocomposite foams (MFCNCF)

FT-IR spectra of pure melamine formaldehyde foam (MF) and MF-organoclay nanocomposite foams (MFCNCF) are given in Figure 4.





In the FT-IR spectrum of the MF-organoclay nanocomposite foam (MFCNCF) in Figure 4, apart from the strong peak at 3354 cm⁻¹ originating from the water-derived hydroxyl group, there are three more peaks at 2926, 2914 and 2881 cm⁻¹, which are the peaks belonging to the secondary amine group stretching, C-H anti-stretching and C–H stretching vibrations.³⁷ All these specific peaks appear at lower intensities and shift to lower values due to interactions between CTA+ ions bound by clay layers and longchain hydrocarbon molecules. In addition, two peaks were observed at 1454 cm⁻¹ and 1129 cm⁻¹, corresponding to the C–N stretching and the N-H bending of CTAB, respectively. On the other hand, it can be claimed that the peaks appearing in the 1302–1657 cm⁻¹ region are due to the CH₂ shift vibration mode and the O-H bending mode of the water molecule around the attached head group. Si-O and Al-OH are the main functional groups observed in the range of 1000 cm⁻¹ to 500 cm⁻¹. The peak at 866 cm⁻¹ corresponds to Al-OH bending vibrations, while the double Si-O-Si bonds in SiO₂ at 802 cm⁻¹ and the Si-O stretching vibrations observed around 714-617 cm⁻¹ indicate the presence of quartz. The emergence of characteristic peaks with low intensity and shifted from their specific values may be indicative of intense interactions between clay plates and CTA+ ions bound to long-chain hydrocarbons.38,39

Characterization of mineralogical structures of melamine formaldehyde foam (MFF)and melamine formaldehyde organoclay nanocomposite foams (MFCNCF)

Figure 5 shows the XRD diffraction patterns of pure melamine formaldehyde foam (MF) and containing different clay ratios MF-organoclay nanocomposite foams (MFCNCF1-3).



Figure 5. XRD diffractograms of pure melamine formaldehyde foam and MF-organoclay nanocomposite foams (MFCNCF1-3) containing different clay ratios.

Figure 5 shows that two typical broad peaks at 9.4° and 23.8° appeared in the XRD pattern of MF foam, indicating an amorphous structure. This means that the formaldehyde resin of melamine is composed of methylol monomers and the polymeric backbone is spread. It can be seen from Figure 5 that the smaller of the two peaks of MF resin partially overlaps with the characteristic smectite peak at 8.1°.^{38,40} This left-shifted and enlarged smectite peak clearly indicates that the polymer molecules can enter the interlayer space of the clay and the nanocomposite is formed. The HRTEM image in Figure 1b also supports this claim.

Thermal conductivities of melamine formaldehyde foam (MFF) and melamine formaldehyde organoclay nanocomposite foams (MFCNCF)

Thermal conductivity is an important indicator for evaluating the thermal performance of a material under constant conditions. Thermal conductivity coefficients measured for different samples were considered in the evaluation of the thermal insulation performance of materials ⁴¹. The thermal conductivity coefficients of pure melamine formaldehyde foam (MFF) and MF-organoclay nanocomposite foams containing different proportions of clay (MFCNCF1-3) are shown in Table 4.

Table 4. Thermal Conductivity Analysis Results of Pure MelamineFormaldehyde Foam and Containing Different Clay Ratios MF-organoclay Nanocomposite Foams

Sample Codes	Heat Conductivity Coefficient (λ) (W/m·K)	Standard Deviation
MFF	0.0826	0.0012
MFCNCF1	0.0869	0.0021
MFCNCF2	0.0650	0.0048
MFCNCF3	0.0812	0.0036

When Table 4 is examined, it is seen that MFCNCF2 has the smallest heat transfer coefficient. This shows that MFCNCF2 can be used as an alternative to insulation materials.

Dependence of density and compressive strength of melamine formaldehyde foam (MF) and melamine formaldehyde organoclay nanocomposite foams on their compositions

Compressive strength is an important mechanical property measured by placing a material directly under compressive loads. Compressive strength is also a critical design feature, and composite materials can exhibit some unique compressive strength behaviors depending on the composite structure. Some materials fracture at the limit of their compressive strength, and some deform irreversibly, so a certain amount of deformation can be considered the limit of the compressive load. Table 5 shows the bulk density and compressive strength values of pure MF foam and MF organo clay nanocomposite foams (MFCNCF1-3).

Table 5. Variation of Density and Compressive Strength ofMelamineFormaldehydeFormaldehydeFoamFormaldehydeOrganoclay NanocompositeFoamsDepending onTheir Compositions

Sample Codes	Bulk Density (g/cm³)	Compressive Strength (N/mm ²)	Standard Deviation
MFF	0.26	0.270	0.002
MFCNCF1	0.39	0.230	0.001
MFCNCF2	0.18	0.320	0.002
MFCNCF3	0.20	0.680	0.007

When Table 5 is examined, it is seen that MFCNCF3 has the highest compressive strength. With increasing clay ratio, the compressive strength has increased approximately threefold. It can be said that the three-fold increase in compressive strength, especially observed at high organoclay ratio, provides increased ductility due to improved adhesion interactions in the brittle MF foam matrix of organoclay plates.

CONCLUSION

This study focused on the synthesis of melamine formaldehyde organoclay nanocomposite foams using microwave irradiation and heating technique together and the investigation of their thermal insulation and mechanical stability.

• Advantages such as faster reaction, high yield and purity, and reduced curing time were obtained with microwave irradiation.

• HRTEM, FTIR, SEM and XRD techniques were used for the characterization of pure melamine formaldehyde foam, MFF and melamine formaldehyde organoclay nanocomposite foams, MFCNCFs, prepared with various organoclay contents. From spectroscopic and microscopic analyses, it was observed that organoclay platelets were exfoliated with increasing clay content without much structural change in the resin matrix.

• The highest compressive strength was obtained in MFCNCF3 foam with high organoclay content (0.68 N/mm²) and it was determined that its bulk density was quite low (0.20 g/cm³).

• In the nanocomposite (MFCNCF2) with 0.15% organo clay content, the compressive strength was measured as 0.32 N/mm² and the thermal conductivity coefficient was measured as 0.065 W/m·K.

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