Investigation of Magnetic Properties of Poly Vinyl Chloride Composites Prepared Using Different Ratios of Fe₂O₃ (Maghemite) Nanoparticles

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Abstract

Composites were prepared using poly vinyl chloride (PVC) and different percentages of Fe₂O₃ nanoparticles. All composites were characterized by FT-IR spectroscopy. Then, the thermal properties of the composites prepared in 1% and 10% ratios were examined. It was observed that the initial decomposition temperature (T_i) values of PVC composites prepared with Fe₂O₃ nanoparticles addition were lower than pure PVC. Magnetic properties of composites were examined with vibrating sample magnetometer (VSM). The saturation magnetization values of PVC composites prepared by using 1% and 10% Fe₂O₃ were 2.77 and 7.06 emu / g, respectively.

Keywords: PVC, Fe₂O₃ nanoparticle, maghemite, VSM, magnetization

Farklı Oranlarda Fe₂O₃ (Maghemit) Nanopartikülleri Kullanılarak Hazırlanan Poli Vinil Klorür Kompozitlerinin Manyetik Özelliklerinin İncelenmesi

Öz

Polivinil klorür (PVC) ve farklı oranlarda Fe₂O₃ nanoparçacıkları kullanılarak kompozitler hazırlandı. Tüm kompozitler FT-IR spektroskopisi ile karakterize edildi. Ardından, %1 ve %10 oranlarında hazırlanan kompozitlerin termal özellikleri incelendi. Fe₂O₃ nanopartikül ilavesi ile hazırlanan PVC kompozitlerin başlangıç bozunma sıcaklığı (Ti) değerlerinin saf PVC'ye göre daha düşük olduğu görüldü. Titreşimli numune manyetometresi (VSM) ile kompozitlerin manyetik özellikleri incelendi. %1 ve %10 Fe₂O₃ kullanılarak hazırlanan PVC kompozitlerin doygunluk manyetizasyon değerleri sırasıyla 2,77 ve 7,06 emu/g bulundu.

Anahtar Kelimeler: PVC, Fe₂O₃ nanopartikül, maghemit, VSM, manyetizasyon

INTRODUCTION

Progress in the synthesis of nanomaterials has led to an increase in research studies on the production and use of nanocomposites (Byrne and Gun'ko, 2010; Thostenson et al., 2001). Nanocomposites have always been the focus of attention for researchers (Tanaydın, 2021). This is because the physical properties of the host materials (usually polymers) match those of nanoscale materials. An example is that a non-magnetic material can become magnetic by adding an appropriate amount of magnetic material such as magnetic nanoparticles (Fe₃O₄, Fe₂O₃) (Wilson et al., 2004). Likewise, the insulating polymer can be given semiconductor or conductive properties by doping

conductive materials (MWCNT, nanographite) (Moniruzzaman and Winey, 2006). Thermal stability is required when polymeric materials are used for a long time and at different temperatures. Thermal stability can be achieved thanks to nanocomposites prepared using inorganic additives in nanosized size (Haruna et al., 2020; Moniruzzaman and Winey, 2006).

Magnetic nanoparticles containing iron oxide (Fe₃O₄ and Fe₂O₃) show superparamagnetic properties. As the size of such nanoparticles gets smaller, superparamagnetism emerges (Berry and Curtis, 2003). Superparamagnetism is a state open to changes in the magnetization direction. Therefore, as

the external magnetic field moves away from the medium, superparamagnetic nanoparticles lose their magnetization properties and return to their initial state (Gossuin et al., 2009; Lu et al., 2007). Magnetic nanoparticles are used in different fields such as magnetic resonance imaging (Lee et al., 2007; Weissleder et al., 2000), drug release (Chouly et al., 1996; Zhang et al., 2002), data storage (Sun Shouheng et al., 2000), catalysis, and energy conversion. The application areas vary according to the controllable size of the particles and the different morphological structures (Shafi et al., 2001). It has been observed in studies that composites prepared by using nanoparticles and especially magnetic iron nanoparticles have improved the properties of polymers such as glass transition temperature, tensile strength, and thermal degradation (Balazs et al., 2006; Haruna et al., 2020; TUKUR et al., 2020).

In order to modify poly vinyl chloride (PVC) and increase its hardness, plasticizers, and fillers were used, and copolymers were prepared using different monomers (Chen et al., 1995; Klarić et al., 2000). PVC is generally chosen as the polymer matrix due to its low cost, biocompatibility, and chemical stability (Haruna et al., 2020). While additives such as carbon fiber (Matuana et al., 1998), wood flour (Djidjelli et al., 2002), and calcium carbonate (Sun Shuisheng et al., 2006) were used to improve the thermal and mechanical stability of PVC, its electrical properties were tried to be improved by using polyaniline (Conn et al., 1995), polypyrrole (Ouyang and Chan, 1996) and MWCNT (Vadukumpully et al., 2011) (Al-Ramadhan et al., 2012). In addition, PVC-membrane electrodes have been developed for ion analysis in ambient water (Adem, 2019).

The homogeneous distribution of magnetic nanoparticles in the polymer matrix is the key to the strengthening and hardening effect of nanoparticles. In this study, PVC / magnetic Fe_2O_3 nanocomposites were synthesized in a micellar environment with Fe_2O_3 nanoparticles as the core in the PVC matrix layer. The characterization of the composites obtained by using nanoparticles on two different percentages (1% and 10%) was made by FT-IR spectroscopy. TGA analysis and magnetic properties of the prepared composites were examined.

Finally, both the saturation magnetization values and particle diameters were calculated from the obtained magnetization curves.

EXPERİMENTAL
Materials
PVC was purchased from PETKIM Turkish Company with a molecular weight of Mn=63000 $gmol^{-1}$. Magnetic Fe_2O_3 nanoparticle (maghemite) and tetrahydrofuran (THF) has been obtained from Sigma-Aldrich.

Preparation of PVC/Fe_2O_3 Nanoparticle Composites
Firstly, we prepared PVC composite with 1% Fe_2O_3 nanoparticle. 0.5 gram PVC was dissolved in 5 mL THF. 0.005 g of Fe_2O_3 nanoparticle was added to PVC solution and was sonicated in an ultrasonic homogenizer for 1h. The resulting composite was precipitated in ethanol and then filtered (Haruna et al., 2020). Using the same procedure, the PVC/Fe_2O_3 composite containing 10% Fe_2O_3 nanoparticle was prepared. Both PVC composites (containing 1% and 10% Fe_2O_3 nanoparticle) were dried first at room temperature and then at 40 °C in a vacuum oven for 24 h (Ma et al., 2010).

RESULTS AND DISCUSSION
FT-IR characterization
FT-IR spectrum of pure PVC, PVC / 1%, and 10% Fe_2O_3 composites are shown in Fig. 1. The characteristic bands of PVC at 2972-2912 cm^{-1} (aliphatic -CH stretching), 1625 cm^{-1} (-OH bending coming from KBr), 1251 cm^{-1} (-CH bending on HCl), and 680 cm^{-1} (C-Cl stretching). In addition, the OH bending vibration at 1625 cm^{-1}, which is not related to the structure, is due to KBr. In the spectrum of PVC / Fe_2O_3 composites, a new peak characterizing the Fe-O bond was detected at 617 cm^{-1} (Pekdemir et al., 2021).

Thermal Properties
TGA curves of PVC / Fe_2O_3 composites are given in Fig. 2. In our previous study (Hassan Haruna, 2019), it was seen that the T_i (initial decomposition temperature) value of PVC was 248 °C. In the case of PVC composites containing 1% and 10% magnetic Fe_2O_3, rapid decomposition began at 213 and 230 °C (T_i), respectively.

It has been observed that the Fe_2O_3 nanoparticles reduce significantly the thermal stability of PVC. Although the thermal stability of the composites increases as the amount of Fe_2O_3 nanoparticles increases, it is seen that they have still lower thermal stability than that of PVC. The results obtained from the TGA curves are shown in detail in Table 1.
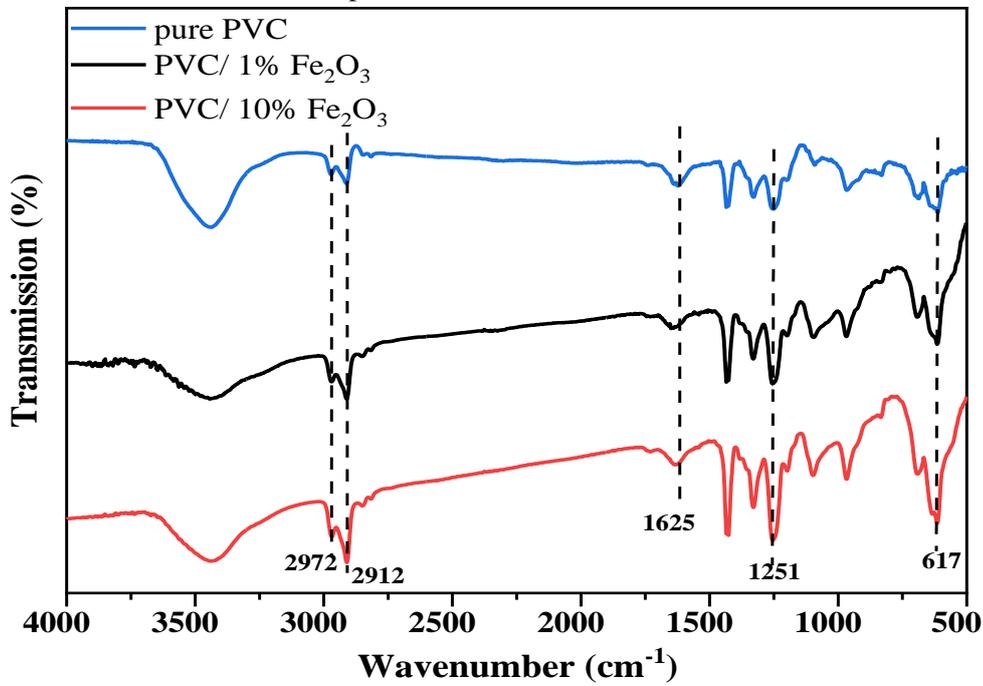

Figure 1. FT-IR spectra of PVC and Fe_2O_3 composite

Table 1. *Thermal investigation of PVC and composites containing Fe_2O_3 nanoparticles at different percentages*

Material	$T_i{}^*$ (°C)	Weight loss (wt) at 350 °C	Residue (wt%) at 500 °C
PVC	250	61.7	10
PVC / %1 Fe_2O_3 composite	215	54.1	12
PVC / %10 Fe_2O_3 composite	230	47.2	16

Magnetic Properties

The magnetic properties of the PVC/Fe_2O_3 composites were characterized by vibrating sample magnetometer (VSM) at 300 K. The most important evidence that PVC composites containing magnetic Fe_2O_3 nanoparticles are the VSM graph given in Fig.3.

It was seen in the VSM curves that the saturated magnetization values (M_s) of the PVC / 1% Fe_2O_3 composite and PVC / 10% Fe_2O_3 composite were 2.77 and 7.06 emu/g, respectively. It is reported that the M_s value of the Fe_2O_3 nanoparticle (maghemite) is 76.0 emu/g (Kommareddi et al., 1996) and 69.7 emu/g (Caizer, 2003). The obtained saturation magnetization values of the composites are lower than the values given for pure Fe_2O_3 due to the organic groups in PVC.

It can be said that the magnetic diameter of the particles is related to the parameters in the equation given below:(Yaacob et al., 1995)
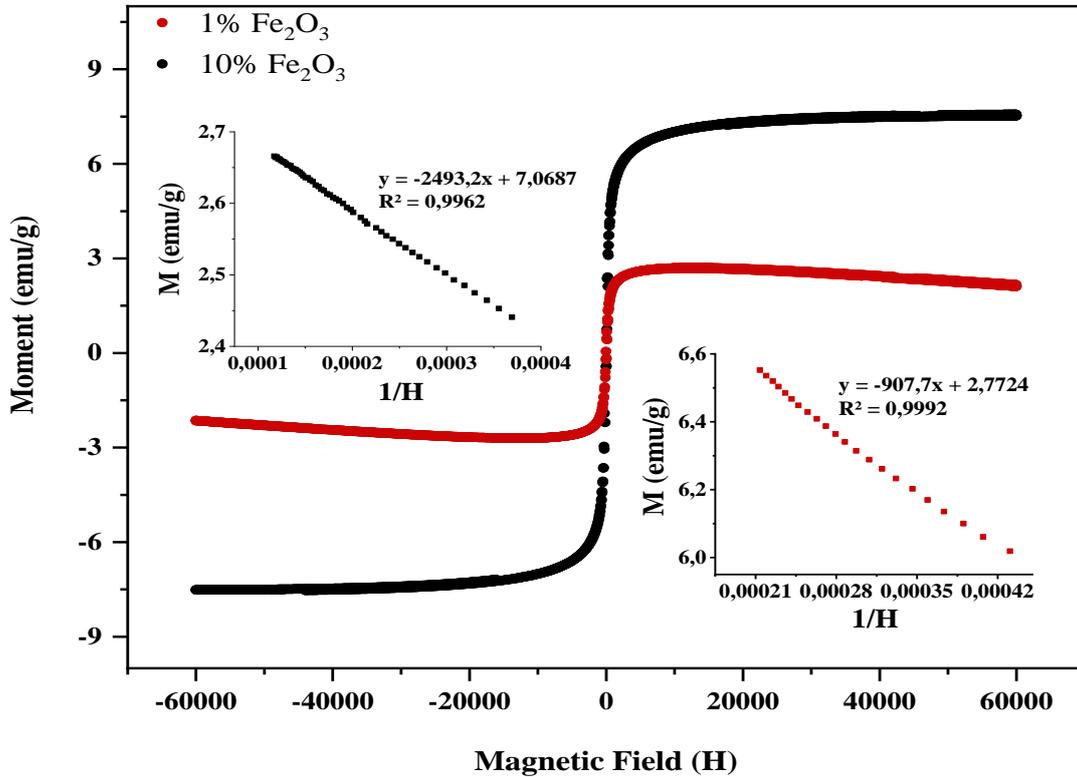
$$d_{mag} = \left(\frac{18kT \left(\frac{d\sigma}{dH} \right)_0}{\pi \rho M_s{}^2} \right)^{1/3}$$

where ρ is the density of maghemite (5.07 g/cm^3)(Kommareddi et al., 1996), k is the Boltzmann constant (1,381x10^{-23} J / particle).
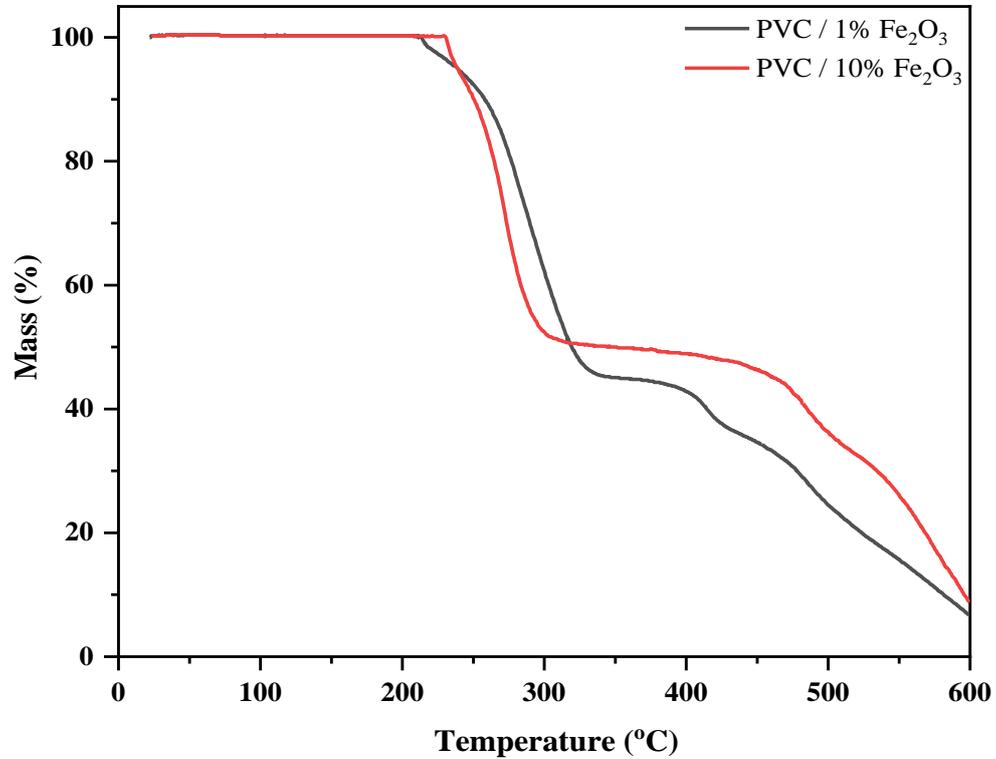
Figure 2. TGA curves of PVC / 1% Fe$_2$O$_3$ and PVC / 10% Fe$_2$O$_3$ composites

Figure 3. VSM plot of PVC/1%Fe_2O_3 and 10% Fe_2O_3 nanoparticle

Magnetic susceptibility values $[(d\sigma/dH)_0]$ for 1% Fe_2O_3 and 10 % Fe_2O_3 composites were calculated as 0.0036 and 0.0019 emu/g.Oe, respectively. The value $(d\sigma/dH)_0$ given in the equation is equal to the magnetic susceptibility (χ_i) (Kommareddi et al., 1996; Yaacob et al., 1995) and is found from the slopes of the tangent line drawn at *H*=0 Oe. Using the equation given above with the magnetization curves obtained by VSM, the particle sizes of PVC composites containing 1% and 10% Fe_2O_3 nanoparticles were calculated as 12.3 nm and 5.3 nm, respectively.

CONCLUSIONS

Composites were prepared with commercially used PVC and 1% and 10% magnetic Fe_2O_3 nanoparticles. In the FT-IR spectra of the composites prepared, the Fe-O stretching vibration at 637 cm^{-1} proved to be nanoparticles in the structure. When TGA curves of composites were examined, it was seen that Fe_2O_3 nanoparticles reduced T_i value, in other words, the thermal stability of composites was lower than pure PVC. In addition, when the magnetization curves are examined, in the absence of an external magnetic field of PVC/Fe_2O_3

nanocomposites, the field strength is required to restore the magnetization at the zero point and the magnetization value at the zero point is insignificant, which means that the composites are superparamagnetic. From the magnetization curves obtained by VSM, the saturation magnetization values of composites prepared with 1% and 10% Fe_2O_3 nanoparticles were 2.77 and 7.06 emu / g, respectively. Therefore, it can be concluded that the composites have superparamagnetic properties.

CONFLICT OF INTEREST

The Author report no conflict of interest relevant to this article.

RESEARCH AND PUBLICATION ETHICS STATEMENT

The author declares that this study complies with research and publication ethics.

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