Effect of Chemical Modification of Oil Fly Ash and Compatibilization on the Rheological and Morphological Properties of Low-Density Polyethylene Composites

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ABSTRACT: In this study, the effect of oil fly ash (OFA), a by-product of oil fuel power plants, on the rheological and morphological behavior of low-density polyethylene (LDPE) is investigated. As received and acid-functionalized OFA (COOH-OFA) are used to examine the effect of surface modification of OFA on polymer–filler composites. LDPE/OFA composites were prepared by melt mixing with filler loading in the range 1–10 wt %. The results are compared with pure LDPE. The effect of polyethylene-*grafted*-maleic anhydride (PE-*g*-MA) as a compatibilizer was also studied. Both viscous and elastic properties of composites increased with OFA loading especially at low frequency. The surface modification of OFA has influenced the properties of OFA. As-received OFA showed some agglomeration at high loading that resulted in two-phase system as described by scanning electron microscopy (SEM) and Cole–Cole plot. Field emission-SEM (FE-SEM) images showed improvement in the dispersion of COOH-LDPE/OFA composites. In addition, the surface modification reduced the size of agglomeration. In general, the COOH modification of OFA improved both the dispersion and rheological properties of OFA. With chemical modification, the concentration of the filler can be increased to 10% without compromising the properties of the composites. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 2486–2496, 2011

Key words: fly ash; chemical modification; dispersion; rheology; polyethylene; morphology

INTRODUCTION

Fly ash is a by-product generated by the combustion of coal and fuel. It is collected by means of electrostatic precipitators or mechanical devices such as cyclones to control the air pollution. It is divided into two main types based on the fuel used for combustion as represented by the names, i.e., "coal fly ash" (CFA) and "oil fly ash" (OFA). According to 2009 survey of American Chemical Society, more than 71 million tons of fly ash produced annually in United Stated by 460 coal-fired plants, and 45% of this quantity was reused in different applications.[1] Mostly, it is used as a replacement of Portland cement, as a filler in polymers, asphalt and cementations materials, stabilization agent, and also for solidification of wastage and sludge. The reuse of fly ash is not only due to its cheap production but also for the protection of the environment. It is estimated by a report of C and EN in December 2009[2] that 7% of global carbon dioxide and greenhouse gases are emitted by cement production, which can be reduced by the use of fly ash. Fly ash is a finely grounded particulate type material mostly composed of unburned carbon, carbon black, and a little amount of sulfur and oxides depending on the fuel used.[3] Some traces of toxic components like arsenic, beryllium, boron, cadmium, chromium VI, cobalt, lead, manganese, mercury, molybdenum, selenium, strontium, thallium, and vanadium may also be present.[4–6]

Polymer composites filled with inorganic mineral fillers had an industrial attraction for their wide applications and low cost. These fillers were used to improve the polymer properties like mechanical strength, rheological behavior, and thermal degradation. Fly ash, clay, silica, and mica are some of the common examples of mineral fillers.[7–10] Uniform distribution of these tiny particles plays an important role in enhancement of properties. It is believed

that the particle distribution in the polymer matrix could be improved by the surface modification of the fillers. It was observed that the attachment of carboxylic acid groups to the different carbon-based materials (such as carbon nanotubes) not only enhanced the interface linking property of the filler but also provided reactive sites to attach a variety of functional groups.[11–14] The application of low-density polyethylene (LDPE) varied from simple shopping bags and toys to bullet proof vests, and it is the most widely used consumer product in the chemical industry. Hence, improvement of mechanical, thermal, and rheological properties of LDPE composites is of interest to the polymer industry. Reuse of OFA would not only reduce the cost of LDPE composite but solve an environmental problem, too.

Many studies showed an increase in viscoelastic properties of polymer–filler composites as compared with pure polymer matrix.[15–19] Increasing the filler content into the polymer matrix changes the polymer behavior from liquid-like to solid-like. The effect of fly ash on polymer rheological properties is discussed in the following section.

Suryasarathi and Mahanwar[20] described the effect of fly ash on mechanical, thermal, dielectric, rheological, and morphological properties of Nylon 6. Fly ash of an average particle size of 8 μm and 60 μm was used in 5–40 wt % ratios. Good dispersion of particles was observed for small particles as compared with large particles in the case of low filler loading (<20%). However, at high loading (>20%) both types showed agglomeration. Yu-Fen et al.[21] investigated the surface modification of fly ash using isothermal heating, and its effect on polymer composites properties was studied. Increase in surface area and whiteness of fly ash particles were observed due to thermal treatment. Also, an improvement in the interface between polypropylene and fly ash was observed after modification. Effect of chemical treatment of surface of fly ash on the properties of natural rubber/fly ash composites was investigated by Thongsang et al.[22] The use of various compositions of bis(triethoxysilylpropyl) tetrasulfane (Si69) for surface modification resulted in increase in the mechanical properties of composites up to 4% loading of the filler. On the other hand, NaOH treatment showed no improvement in properties.

Wu et al.[23] studied the influence of chlorinated polyethylene (CPE) on mechanical properties, morphology, and rheology of nanocomposites of poly (vinyl chloride) (PVC) and nanometric calcium carbonate particles. The rheological study showed a remarkable increment in melt viscosity by the addition of $CaCO_3$ nanoparticles in PVC, whereas the viscosity decreased by the addition of CPE. Transmission electron microscopy (TEM) results showed $CaCO_3$ particles dispersed uniformly through the matrix of PVC. The rheological properties of polystyrene/layered silicate nanocomposites are investigated by Lim and Park.[24] Authors showed that both storage and loss moduli increased with silicate loading at all frequencies and showed nonterminal behavior at low frequencies. Further, McNally et al.[25] reported the rheology of polyethylene (PE) multiwall carbon nanotubes (MWCNTs) composite with filler loading ranged from 0.1 to 10. Dynamic viscosity (η'), storage modulus (G'), and loss modulus (G'') increased with the increase of the concentration of MWCNT and results more dominant at low frequencies. Also, the low frequency was suggested to indicate "pseudosolid-like" behavior.

The aim of the current study was the investigation of the effect of chemical modification of OFA on the rheological and morphological properties of LDPE composites. Four different loadings of OFA in the range of 1–10 wt % were used. Also, the effect of polyethylene-*grafted*-maleic anhydride (PE-*g*-MA) as a compatibilizer was examined.

EXPERIMENTAL

Materials

OFA was supplied by Saudi Aramco and produced by local power generation plants. The Energy dispersive X-ray analysis (EDXA) was carried for the analysis of chemical composition of OFA. The chemical composition of as-received OFA is as follows: 92.5% carbon; 5.80% sulfur; 0.79% magnesium; 0.09% silicon; and 0.61% vanadium. This analysis shows that the OFA used in this research was mainly composed of carbon material with a very little amount of sulfur. Also, the presence of some heavy metals was observed in traces, which is typical for heavy oil combustion residue. Sulfuric and nitric acids used for the surface modification of fly ash were produced by Sigma Aldrich Company (St. Louis, MO). Sulfuric Acid has a purity >95%, and a density of 1.83 g/mL, whereas nitric acid has a purity >90%, and a density of 1.48 g/mL. De-ionized water with a pH ~7.0 was used for washing glassware and filtration of product.

LDPE was supplied by Nova Chemicals, Canada. The LDPE has a weight average molecular weight of 99.5 kg/mol, and a molecular weight distribution (MWD) of 6.5, and a melt index of 0.75 g/10 min, and a total short branch content of 22 branches/1000 C as determined by gel permeation chromatography (GPC) and nuclear magnetic resonance (NMR), respectively (Hussein and Williams).[26] The PE-*g*-MA was received by Sigma Aldrich, and it has a viscosity of 1400–1700 cP, a density of 0.925 g/cm^3, and a melting point of 105°C.

Surface modification of fly ash

Surface treatment of dried OFA was done with a mixture of sulfuric acid and nitric acid.[27] The
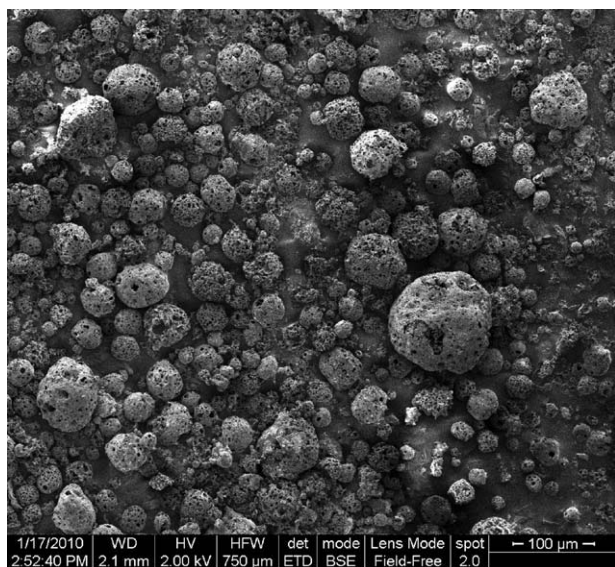
Figure 1 Fly ash grains at magnified view.

heating of acid–ash mixture started at a temperature of 150–160°C with continuous stirring by glass rod. Also, the air flow was used to enhance the oxidation of OFA particles. The product was then washed after cooling down to room temperature. Hot de-ionized water was used for washing of the modified OFA using Buchner funnel to remove all un-reacted acid present in the product. After washing, wet OFA was dried into the oven at a temperature of 105°C for 1.5 h. The final product was OFA functionalized by carboxylic group. Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and spot analysis was carried out to confirm the attachment of carboxylic acid functional groups to the surface of OFA.[27] Figure 1 shows the fly ash grains, as obtained by SEM in the current study. Figure 2(a,b) shows the FTIR spectra of OFA before and after acid treatment.

Preparation of LDPE/OFA composites

Preparation of LDPE/OFA composites was performed in a Brabender Melt Mixer. The blender consists of three heating zones with independent temperature control systems. LDPE/OFA composite samples were prepared with four different OFA concentration (1, 2, 5, and 10 wt %). PE-*g*-MA was used as a compatibilizer to enhance the dispersion and compatibility between LDPE and OFA. Physical premixing of LDPE, OFA, and compatibilizer was done into a beaker. Then the composites were fed to a Brabender Melt Mixer at a temperature of 190°C, and a blending speed of 50 rpm. The time of melt mixing for each sample was 10 min. Sixteen composite samples were prepared with different compositions. For the first four samples, the effect of filler concentration of unmodified OFA was examined without the addition of the compatibilizer (control samples). Then another

set of four samples were used to study the influence of the compatibilizer in the absence of chemical modification. Then the same process was repeated for chemically modified OFA/LDPE composites.

Characterization

Rheological analysis

For rheological analysis, samples were prepared using a Carver press. Advanced rheometrics expansion system (ARES) controlled strain rheometer was used for rheological testing. Dynamic shear tests were carried out using cone-and-plate geometry. A plate of 25 mm diameter and cone of 0.1 radians angle and 0.048 mm gap were used. A strain sweep test was performed to select a strain in the linear viscoelastic range. Strain of 15% was used at a temperature of 190°C, and the frequency was varied from 100 to 0.01 rad/s.

FE-SEM analysis

Scanning electron microscopy (SEM) of polymer composites was conducted to observe the morphology of
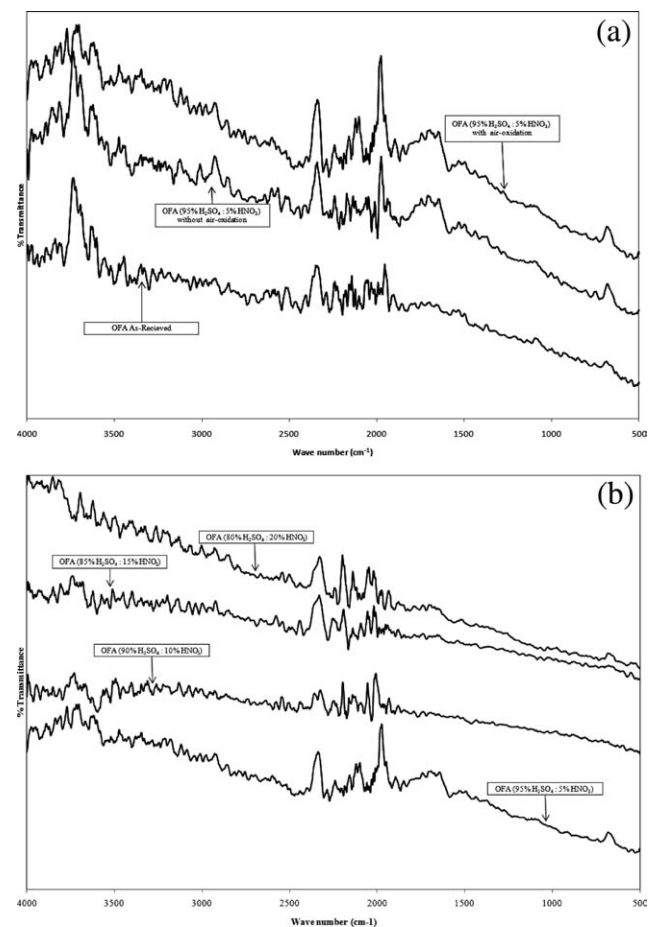

Figure 2 (a) FTIR spectra of as-received and modified OFA with and without air oxidation. (b) FTIR spectra of OFA modified by different acid composition and air oxidation.
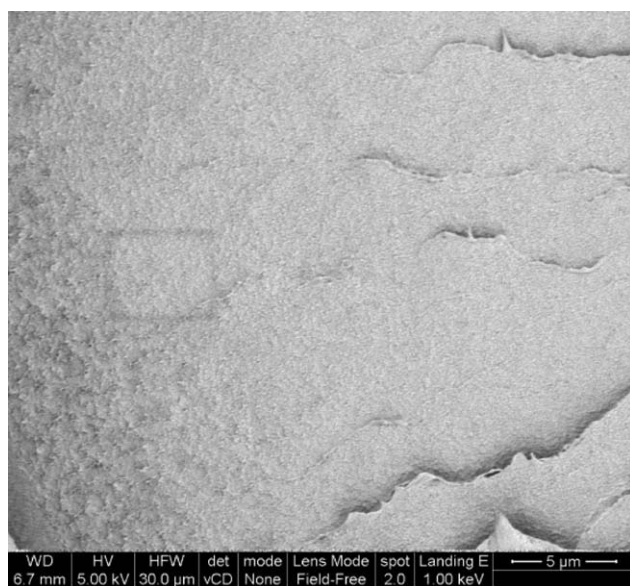
Figure 3 SEM for pure LDPE at 5 μm scale.

Figure 4 SEM for LDPE/OFA composite at 2% loading and 5 μm scale.

LDPE/OFA composite and dispersion of OFA into the LDPE matrix. Fresh surface of samples was prepared to remove any impurity or particle from the surface. For this purpose, the samples are dipped into liquid nitrogen for 1 min and then quickly broken. These broken samples were subjected to nanoscaled gold coating. The gold coating was done for 20 s using sputtering machine. Field emission-SEM (FE-SEM) analysis was done at different resolutions ranging from 500× to 40,000×.

RESULTS AND DISCUSSIONS

Morphological analysis

The FE-SEM analysis of pure LDPE sample was done as a reference as shown in Figure 3. The dispersion of OFA at 2% and 5% loading is shown in Figures 4 and 5, respectively. It is observed that the OFA particles are distributed well in the polymer matrix. However, some agglomeration of particles is also observed. The dispersion of surface modified OFA particles at 2% and 5% loading is shown in Figures 6 and 7, respectively. A better dispersion of OFA particles is observed after the surface modification as compare to unmodified OFA. A well-dispersed acid-functionalized OFA (COOH-OFA) particles were observed at high and low magnification. The degree of dispersion is improved by acid functionalization of OFA as reflected in the reduction of the size of agglomeration. A comparison of results for 2% OFA shown in Figures 4 and 6 suggests a reduction of particle size by a factor of at least 2. Similarly, a comparison of Figures 7 and 5 suggests improved dispersion at 5% OFA loading as
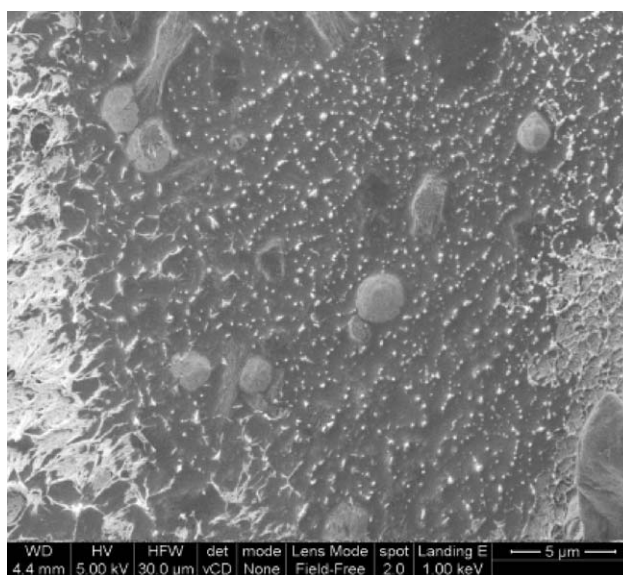
a result of surface modification. All of these comparisons were made for images of the same magnifications. However, the COOH modification of OFA did not eliminate the agglomeration but rather reduced its size as shown in Figures 4 and 6. Similar results are observed when PE-*g*-MA was used as compatibilizer.

Degree of dispersion by Cole–Cole plot

The relaxation behavior of polymer composites can be represented by Cole–Cole plots, which depend on
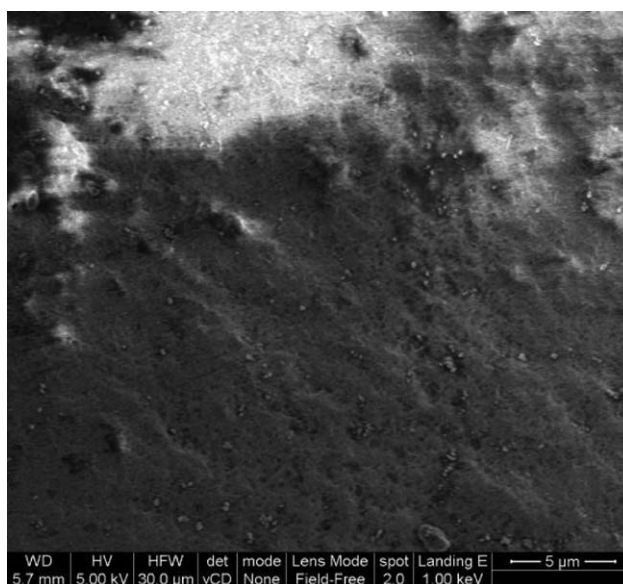

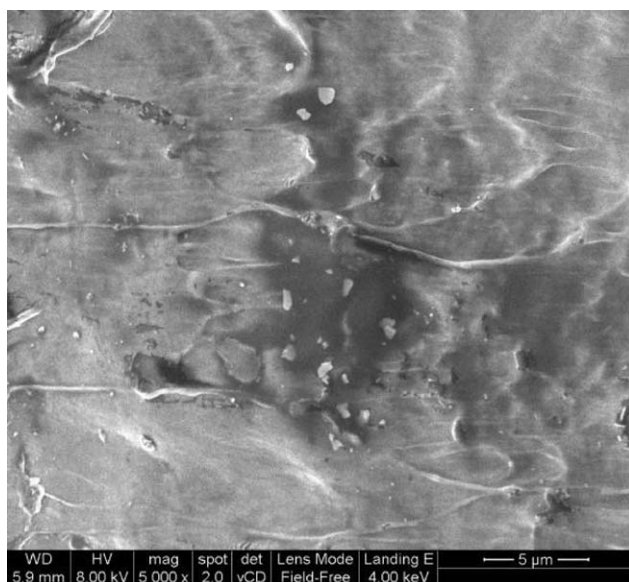**Figure 5** SEM for LDPE/OFA composite at 5% loading and 5 μm scale.

Figure 6 SEM for LDPE/COOH-OFA composite at 2% loading and 5 μm scale.

the degree of dispersion of the filler. It is a plot of imaginary viscosity (η'') versus real viscosity (η'). The high the degree of dispersion results a longer relaxation process. Figure 8(a) shows the Cole–Cole plot of LDPE/OFA composites at different filler loading. As shown, for pure LDPE the relaxation process was short. The mean relaxation time is given by the inverse frequency in which the maximum in Cole–Cole plot occurs.[28–32] For 1% and 2% filler loadings, the degree of dispersion increased with filler concentration but at 5% and 10% loading, it again decreased. It means that the agglomeration has
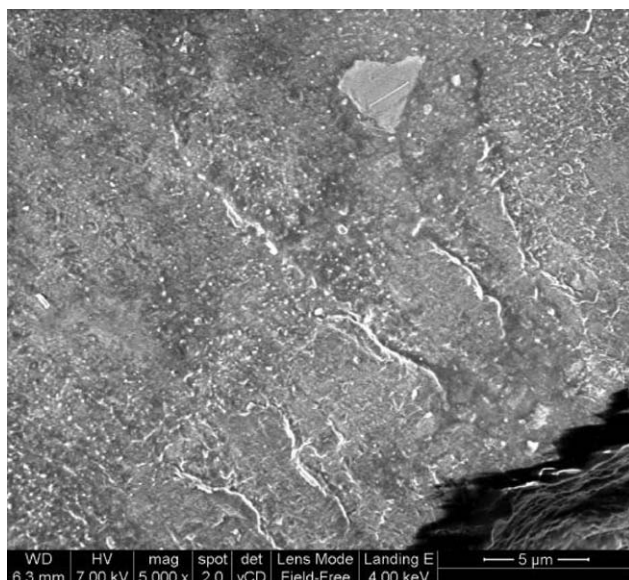

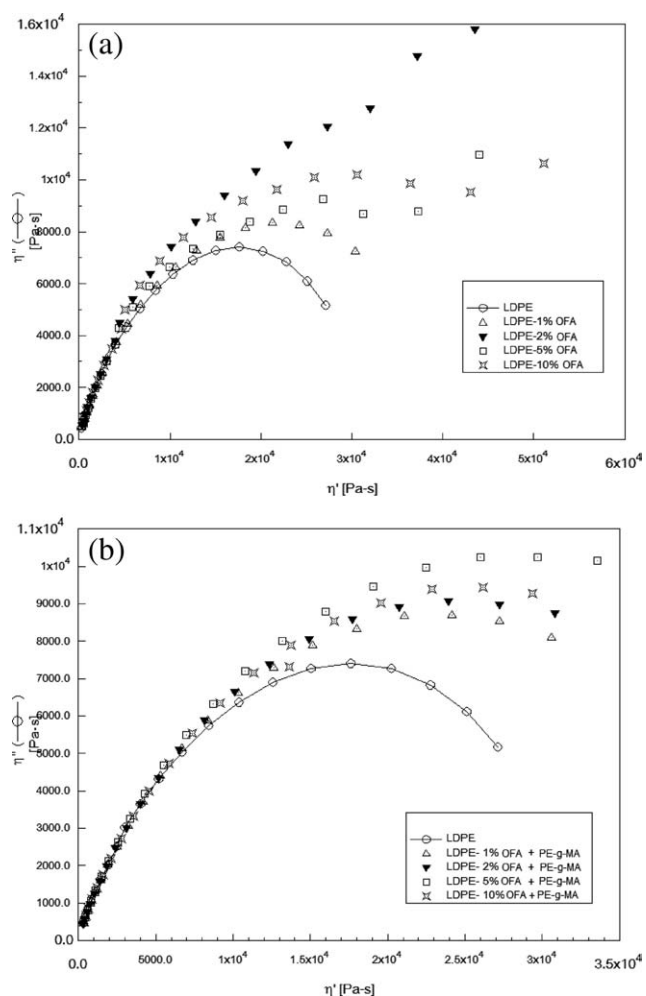**Figure 7** SEM for LDPE/COOH-OFA composite at 5% loading and 5 μm scale.

Figure 8 (a) Cole–Cole plot for LDPE/OFA composite. (b) Cole–Cole plot for LDPE/OFA composite with PE-*g*-MA.

occurred at higher filler loading in the case of as-received OFA. Also, the tail in the 5% and 10% OFA composites indicates the presence of two relaxation mechanisms because of nonhomogeneous behavior.[33,34] Figure 8(b) shows the Cole–Cole plot of LDPE/OFA composite with 2% PE-*g*-MA. The degree of dispersion of OFA has improved in the presence of the compatibilizer and maximum relaxation time observed at 5% filler loading. The shape of the Cole–Cole plot suggests a droplet morphology[35] which is supported by our previous SEM results.

The Cole–Cole plot of COOH-OFA and LDPE polymer composite is shown in Figure 9(a). It is observed that in each case, the relaxation time increased with the addition of filler. The highest degree of dispersion is observed at 5% loading and then decreased at 10% loading due to the increase in the size of agglomerate. Also, the high relaxation time as compared with OFA composites shows a good dispersion of filler due to functionalization. The effect of PE-*g*-MA compatibilizer on the degree
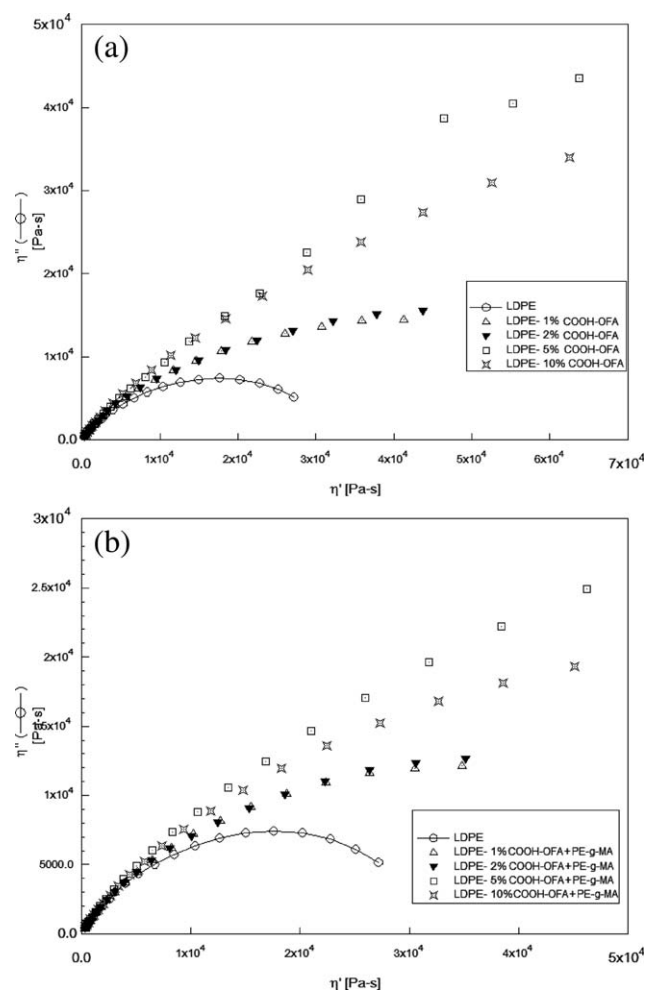
Figure 9 (a) Cole–Cole plot for LDPE/COOH-OFA composite. (b) Cole–Cole plot for LDPE/COOH-OFA composite with PE-*g*-MA.

of dispersion of functionalized OFA is shown in Figure 9(b). A slight improvement is observed after the addition of PE-*g*-MA at high loading.

Effect of modification and OFA loading on storage modulus

The storage modulus, G', basically represents the stored energy of the elastic portion of viscoelastic materials. Figure 10(a) shows the effect of as-received OFA loading on G' of LDPE composites. G' increased at all loadings, and the increase is more dominant at low frequency.[28,29] At low frequency, G' increased linearly for 1% and 2% OFA but dropped down at 5% and 10% loading. This suggests a poor distribution or agglomeration of OFA at high loadings. However, at high frequency, the rheology of LDPE composite is insensitive to the OFA loading.

Figure 10(b) shows the effect of COOH-OFA loading on G' of LDPE composites. As the concentration

of COOH-OFA in the polymer matrix is increased, G' increased, and the composite shows more solid-like behavior. The increase is more dominant at low frequencies. Also, at low frequency, 1% and 2% loadings of COOH-OFA show almost the same degree of improvement. The maximum increase is observed at 5% loading; however, a slight decrease was obtained at 10% loading. At high frequency, results for as-received and modified OFA are similar.

The effect of the use of 2% PE-*g*-MA as a compatibilizer on G' of LDPE composites with OFA and COOH-OFA is shown in Figure 10(c,d), respectively. For as-received OFA, G' increased linearly with the addition of filler up to 5% filler loading then it slightly decreased at 10% loading. The shift in the increase in G' with COOH-OFA loading from 2% to 5% suggests a positive role for the chemical modification. These results provide evidence of good dispersion of OFA at low and moderate loadings which is supported by the previous SEM results. Whereas, poor distribution was obtained at high loading (10%) even in the presence of a compatibilizer. For as-received OFA and PE-*g*-MA, at all loadings almost similar results were observed with little improvement in G' over pure LDPE [Fig. 10(c)]. On the other hand, COOH-OFA system [Fig. 10(d)] showed more improvement in G' of the composites as well as distinct increase in G' with loading. The increment is more dominant at low frequency as reported earlier.

A comparison of G' for modified and unmodified OFA at 5% loading is shown in Figure 11(a). The functionalized OFA showed higher values of G' as compared with untreated OFA. This is a clear evidence that the chemical modification of OFA resulted in good dispersion of OFA in the polymer matrix even at very high loading. This dispersion is not achieved in the case of as-received OFA. Similar results are obtained at 1, 2, and 10 wt % loadings, and the difference is getting more pronounced with the increase in filler concentration. The enhancement in elastic modulus of LDPE composites is mainly at low frequency. The effect of PE-*g*-MA compatibilizer on G' with OFA and COOH-OFA at 5% loading is shown in Figure 11(b,c), respectively. The addition of the compatibilizer did not result in the increase in G' of the composite, in the case of unmodified OFA. However, the modified OFA showed increase in G' at low frequency. Similar behavior is obtained at 1%, 2%, and 10% loadings. The effect of compatibilizer can be easily observed at almost the entire range of frequency, and it is more pronounced in the case of COOH-OFA. These results suggest the impact of chemical modification of OFA on G' dominates the influence of the compatibilizer as shown by Figure 11(c).
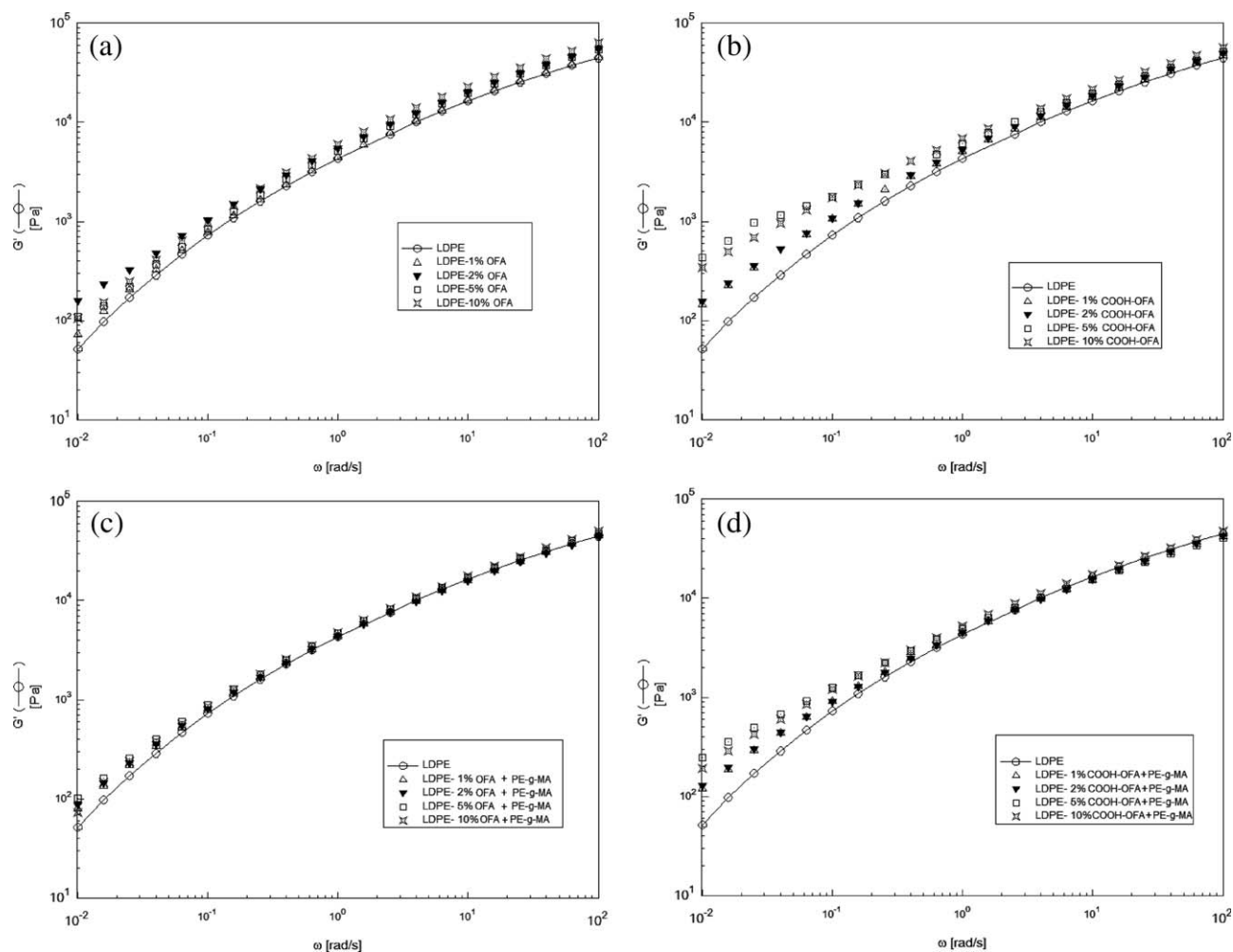
Figure 10 (a) Effect of OFA loading on storage modulus. (b) Effect of COOH-OFA loading on storage modulus. (c) Effect of OFA loading with 2% PE-g-MA on storage modulus. (d) Effect of COOH-OFA loading with 2% PE-g-MA on storage modulus.

Effect of OFA modification and loading on loss modulus

The loss modulus, G'', is directly related to the dissipated energy of the material as it is the amount of energy lost due to viscous flow. Figure 12(a) shows the effect of OFA loading on G'' of LDPE composites. It is shown that G'' increased with OFA loading especially at low frequency.[28,29] All compositions showed similar increase in G'', i.e., the OFA loading had no influence on G'' for untreated ash. However, for modified OFA the 1% and 2% loadings showed similar results. Also, the 5% and 10% loadings showed similar results but higher than those of 1% and 2%. It is suggested that the poor distribution of OFA at such high loadings is the reason behind these observations similar to the previous results of G'.

Figure 12(b) shows the effect of COOH-OFA loading on G'' of LDPE composites. The results show that G'' increased with the increase in the concentration of filler into the polymer matrix. The increment

rises linearly with the OFA loading. Almost similar results are obtain at 1% and 2% loading and reach to maximum enhancement at 5% filler concentration. A very slight decrease is observed at 10% loading. The results of G'' support the previous findings from G'. Figure 12(c) shows the effect of OFA and PE-g-MA as compatibilizer on G'' LDPE composites. No considerable change is observed in the values of G'' in the case of OFA loading in the presence of the compatibilizer. When functionalized OFA was used with PE-g-MA, the increase in properties is observed at low frequency. The effect of functionalization on the properties is shown in Figure 12(d). Again, there is no effect for the compatibilizer on filler dispersion at very high filler loadings.

A comparison of G'' for modified and unmodified OFA at 5% filler loading is shown in Figure 13(a). Results show that the functionalization of OFA has a clear impact on the enhancement of G''. At each loading, COOH-OFA displays higher values of G'',
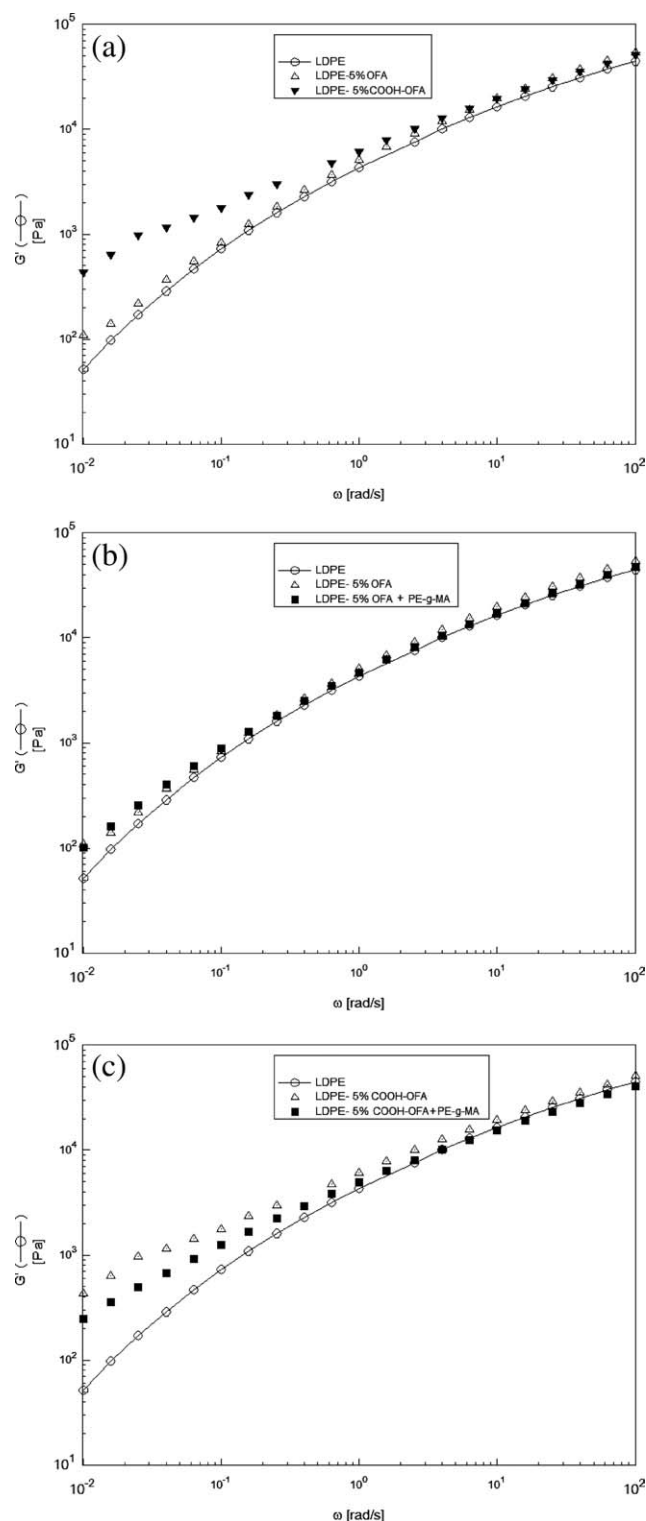
Figure 11 (a) Effect of OFA functionalization at 5% loading on storage modulus. (b) Effect of PE-*g*-MA at 5% OFA loading on storage modulus. (c) Effect of PE-*g*-MA at 5% COOH-OFA loading on storage modulus.

and the difference is more pronounced with the increase in the concentration of OFA. It is a direct result of good dispersion of COOH-OFA as proved by FE-SEM results discussed earlier. Similar behav-

ior was obtained at higher loadings, which is not the case for as-received OFA. At high frequency, very minor decrease in G'' is observed in the case of COOH-OFA, which suggests improved flow behavior of polymer composites.[28,29]

The effect of the compatibilizer on as-received and COOH-OFA at 5% filler loading is shown in Figure 13(b,c), respectively. The addition of the compatibilizer resulted in a decrease in G'' of the composite in both cases due to the low viscosity of the compatibilizer as compared with pure LDPE. The effect of compatibilizer is more dominant in the case of functionalized OFA, and the difference is more obvious at high filler loading as compared with low loadings. At high frequency, G'' for the compatibilized COOH-OFA was lower than G'' for pure LDPE. This observation indicates that the COOH functionalization acts as reinforcement but leads to slight shear thinning at high frequency. The synergistic effect of the compatibilizer, and the functionalization is expected to ease the processing of these composites. Similar behavior is observed at 1%, 2%, and 10% filler loading.

Crossover point and crossover frequency

Crossover point, G_c, is the intersection point of G' and G'' curve when plotted against frequency. The frequency at which this intersection occurs is called crossover frequency, ω_c. Crossover point defines the separation of elastic and viscous behavior of the material. At this point, ω_c represents the homogeneity of the composite material. The low value of G_c and ω_c suggests that the material is more homogenous due to fine dispersion.[30–36] In the case of as-received OFA, G_c and ω_c increased with the increase in filler loading for unmodified fly ash as shown in Figure 14(a). It shows the poor distribution of filler within the polymer matrix and phase separation at higher loading due to agglomeration of filler particles. However, in the case of COOH-OFA, G_c and ω_c decreased with the addition of modified ash. The slight increase in G_c at 10% loading is consistent with our previous observations from Cole–Cole plot that showed slight drop in the curve for the 10% OFA loading. This suggests the fine and constant degree of distribution of COOH-OFA in the polymer matrix.

The effect of filler loading on G_c and ω_c can be described more precisely by Figure 14(a,b). In Figure 14(a), with the addition of the compatibilizer the distribution of as-received OFA has improved hence G_c and ω_c decreased at low loadings. For COOH-OFA composites shown in Figure 14(b), both G_c and ω_c decreased indicating the improvement in the dispersion. So, in the absence of functionalization, the compatibilizer tends to reduce G_c and ω_c, which suggests improvement of dispersion. However, in the case of
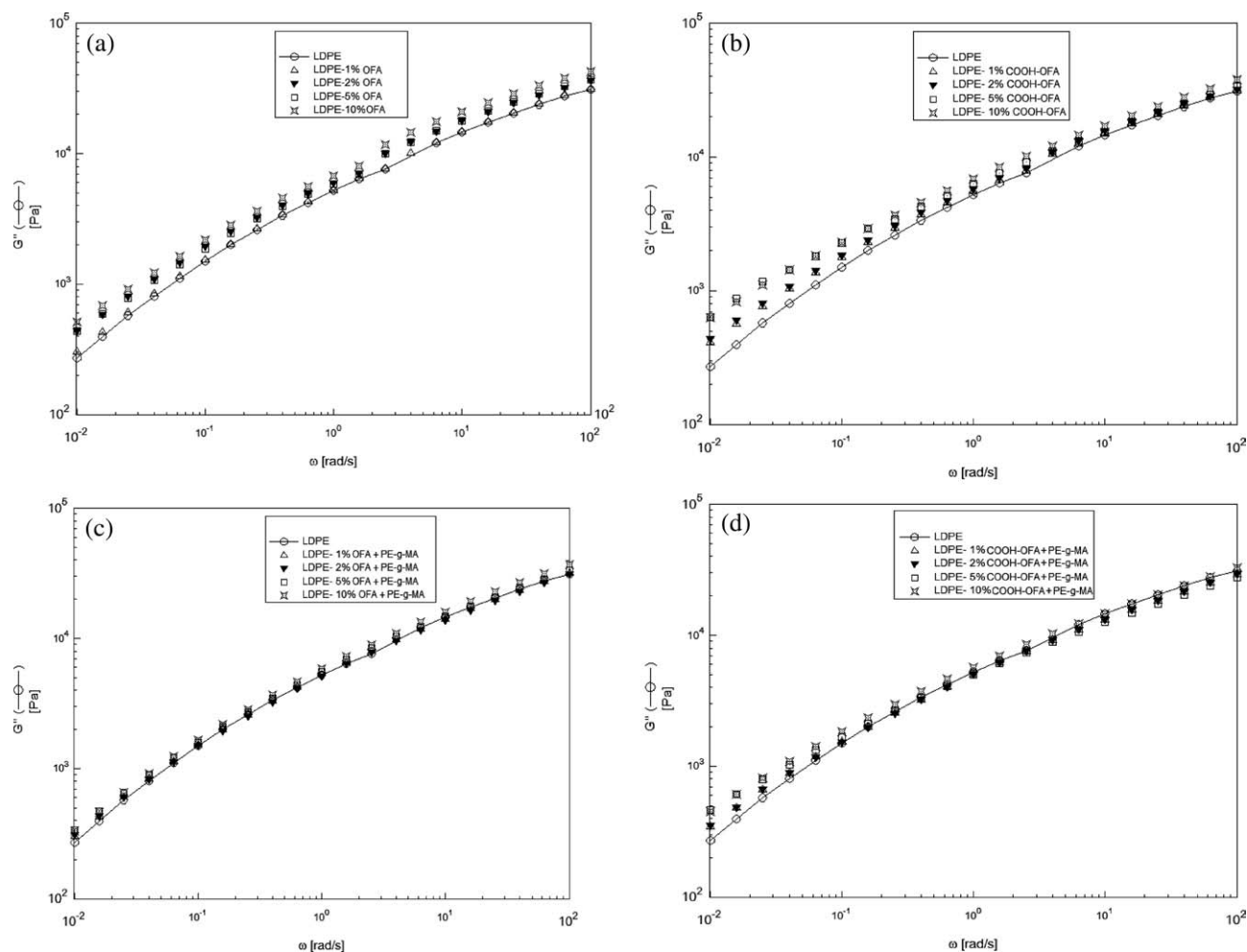
Figure 12 (a) Effect of OFA loading on loss modulus. (b) Effect of COOH-OFA loading on loss modulus. (c) Effect of OFA loading with 2% PE-*g*-MA on loss modulus. (d) Effect of COOH-OFA loading with 2% PE-*g*-MA on loss modulus.

functionalized OFA, the role of the compatibilizer is, in general, not significant or negative [see Fig. 14(b)].

CONCLUSIONS

In this study, LDPE/OFA composites were prepared by melt-mixing method, and rheological and morphological properties were investigated. Surface modification of OFA is performed to enhance the polymer–filler interaction. Also, the effect of PE-*g*-MA as a compatibilizer was studied. Here are the main conclusions of this research:

- FE-SEM analysis showed improved dispersion of OFA in the case of chemically modified ash as compared with unmodified ash. Aagglomeration is observed in the SEM images of LDPE/OFA composites.
- An increase in storage modulus, loss modulus is observed as a result of the addition of ash, especially at low frequency.

- In the case of OFA, good dispersion is achieved at 1% and 2% filler loading, but some agglomeration is observed at 5% and 10% loadings. However, the COOH-OFA showed improved dispersion even at high loadings. It shows that the polymer–filler interaction increased due to the surface modification of the filler. With chemical modification, the concentration of the filler can be increased to 10% without compromising the properties of the composites.
- Crossover point and crossover frequency increased with filler loading in the case of OFA, whereas it decreased after surface modification of filler. It is a clear evidence to better dispersion of COOH-OFA as compared with as-received OFA.
- Cole–Cole plots show two-phase system due to agglomeration of fillers at 5% and 10% loading of OFA. However, improved dispersion is suggested by Cole–Cole plots in the case of COOH-OFA and confirmed by SEM.

- Addition of PE-g-MA enhanced the polymer–filler interaction of unmodified fly ash. However, its impact on modified OFA as in not significant.
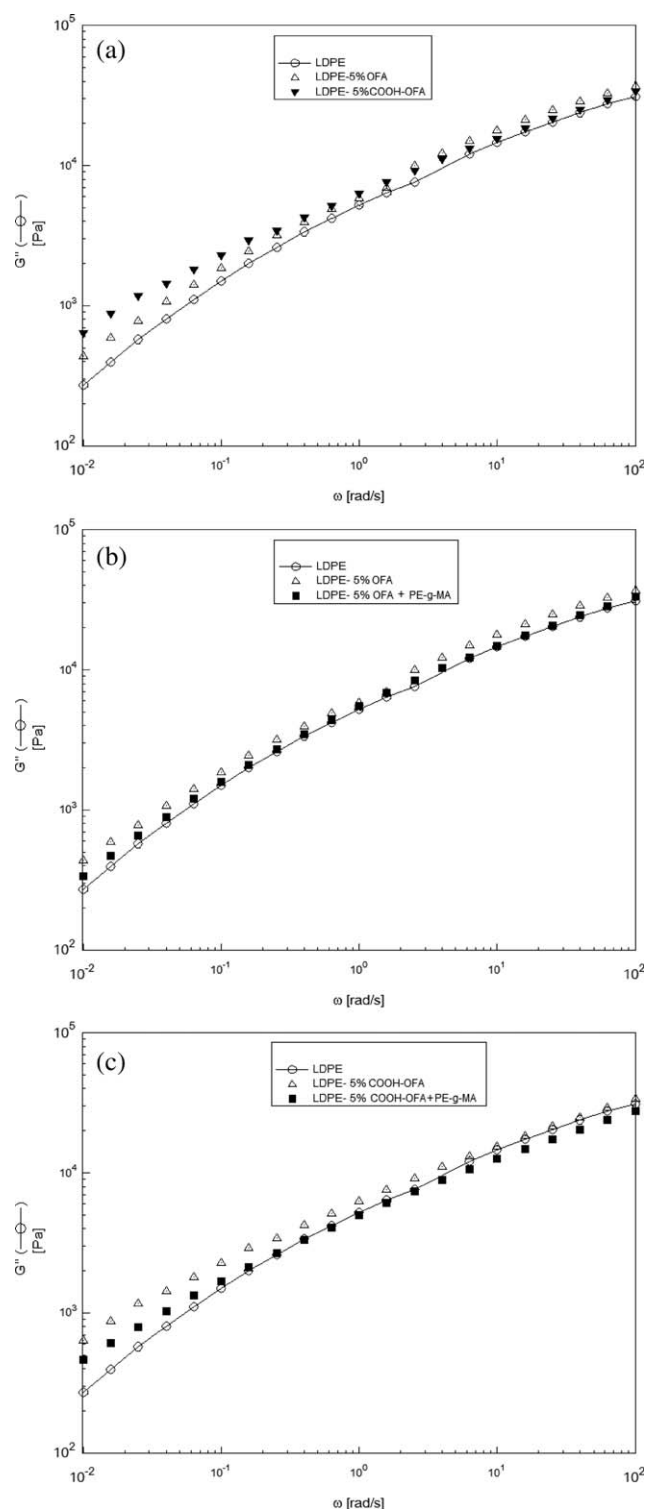

Figure 13 (a) Effect of OFA functionalization at 5% loading on loss modulus. (b) Effect of PE-g-MA at 5% OFA loading on loss modulus. (c) Effect of PE-g-MA at 5%COOH-OFA loading on loss modulus.
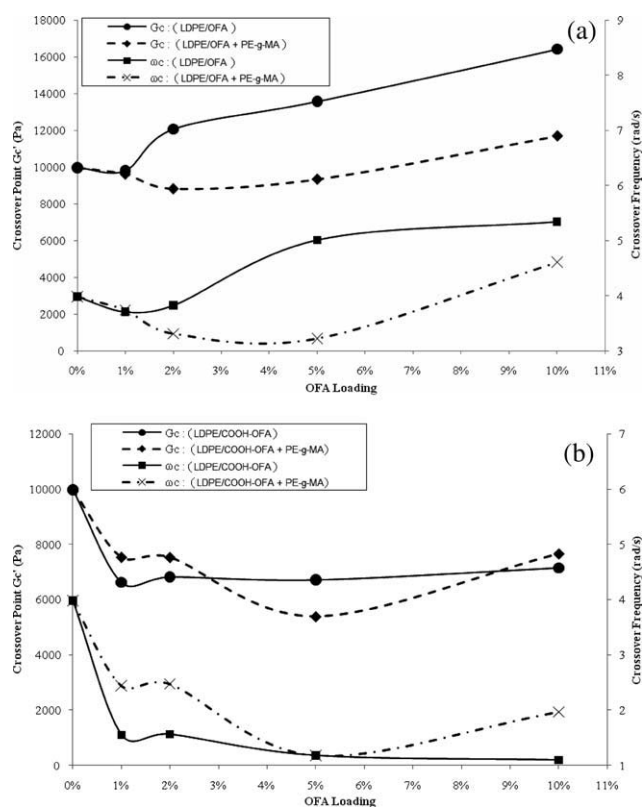

Figure 14 (a) Effect of OFA Loading on G_c and ω_c. (b) Effect of COOH-OFA loading on G_c and ω_c.

From the above results, it is concluded that OFA can be used as a filler to enhance the properties of LDPE and reduce the amount of polymer in the composites. Finally, the use of chemically modified OFA proved to be very useful for the improvement of the properties of PE composites up to 10% by weight. The use of such waste material is expected to reduce the amount of polymer in the composite and help clean the environment from industrial wastes. However, the impact of processing conditions on the dispersion and final properties of these composites need to be studied.

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