Modification of Polyethylene Surface Using Plasma Polymerization of Silane

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ABSTRACT: In the present study plasma polymerization of tetraethylorthosilicate and hexamethyldisiloxane was carried out on polyethylene film with an aim of enhancing barrier properties. The glow discharge was obtained at 0.2 mbar using 13.56 MHz radio frequency source capable of giving power out put up to 100 W. The monomer vapors were passed in the system at the rate of 15 SCCM. The extent of deposition was determined from the weight gain study and the morphology was studied using SEM. Films were further characterized by surface energy measurement, ATR-FTIR and ESCA analysis. Since PE is widely used for packaging and in this process a thin layer of glass-like transparent SiOx was deposited, therefore it was thought necessary to study permeability to water vapors. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 4075–4082, 2007

Key words: polyethylene; plasma polymerization; TEOS; HMDS; ESCA; ATR-FTIR; SEM

INTRODUCTION

Polymers with well-defined functional surface properties are of interest in recent technology.[1] Many attempts, involving physical and chemical interactions, have been made to improve the surface chemical and mechanical properties of polymer.[2] Plasma polymerization technique is considered to be a unique method of preparation of thin polymeric films and the application of this technique to several fields has been studied by various researchers.[3–6] The polymeric films produced by this process usually have highly branched and crosslinked chemical structures and are difficult to dissolve in the usual organic solvents.[7] Recently these films are used for applications such as protective coatings for optical,[8] barrier films for food and pharmaceutical packaging, corrosion protection layers,[9] coatings for biocompatible materials,[10] sensors,[11] and microelectronic components.[12] Because SiOx films are chemically inert, transparent, and dense, they exhibit properties that could make them successful in reducing chain mobility of polymer surfaces.[13] Polymeric packaging materials with good oxygen-gas barrier properties are sought for protecting food or medicines from deterioration of their quality by oxidation. SiOx coated biaxially stretched polyethylene (PE) films are possible materials for this purpose. Plasma processing of organosilicon compounds have been carried out with the aim of improving certain properties such as high thermal stability, dielectric properties, and good scratch resistance.[8] On the other hand plasma treatment of polymers using reactive and nonreactive gases have been carried out to modify surface morphology and surface energy.[14] Little attention has been devoted on the plasma polymerization for improving barrier properties of polymeric films. Keeping this in mind we have studied the effect of plasma deposited siloxane coating on the barrier properties of PE film. Plasma method is a good process for preparing SiOx-deposited PE film because the synthesis of SiOx and the coating of the PE film with SiOx occur simultaneous in one process. Another advantage is that the reaction is carried out at ambient temperature, which is good for substrate polymer like PE having glass transition temperature $\sim$ 70–80°C. Inagaki et al.[15] have successfully deposited SiOx layer on PET substrate with a precursor tetramethoxysilane (TMOS), and showed the effectiveness of plasma polymerization technique for the deposition of SiOx layer on polymers where is T_g is $\sim$ 70–80°C. In the present study, vapors of tetraethylorthosilicate (TEOS) and of hexamethlydisiloxane (HMDS) were allowed to pass into the plasma reactor for polymerization. To have sufficient vapor pressure, TEOS and HMDS reservoirs was kept at 80 and 50°C temperature, respectively. The monomer flow rate was adjusted to 15 SCCM.

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EXPERIMENTAL

Materials

PE films were supplied by Reliance India of thickness of 40 µm. Before treatment the films were cleaned with acetone in an ultrasonic bath for 6 min and then dried in air. PE films were partially crystalline in nature. The monomers TEOS and HMDS were obtained from MERCK India.

Weight gain

Weight gain is determined by the following formula

$$\text{Weight gain} = \{(W - W_o)/W_o\} \times 100$$

where W_o is the initial weight and W is the weight after deposition.

The details of the plasma processing chamber has been reported else where.[14] Plasma reactor was first evacuated to 0.005 mbar pressure and then monomer vapors was passed and working pressure was adjusted to 0.2 mbar. The two electrodes were capacitively coupled to the RF source capable of giving power output up to 100 W. The time of treatment was varied from 5 to 20 min.

The Surface Energy (SE) was calculated by measuring the angle of contact using sessile drop method. Contact Angle (CA) was measured wrt four different liquids: Water (W), Glycerol (G), Formamide (F), and Ethylene Glycol (E.G.) of known polar (γ_L^p) and disperse (γ_L^d) components. The SE was calculated from contact angle measurement using Fowkes approximation. The detailed calculation is given elsewhere.[14] At least 10 reading were taken at different places and an average value was determined.

The ATR-FTIR spectra were recorded by using a Perkin–Elmer Paragon 500 FTIR spectrometer. A KRS-5 crystal with an angle of incidence of 45° was used for recording the ATR spectra. ATR spectra were recorded immediately after plasma polymerization. For every ATR spectrum 64 scans were taken with a resolution of 4 cm^{-1}. Since the PE is widely used by packaging industries for packing food and medicine, it was thought interesting to study the permeability characteristics of plasma processed PE films. For this purpose, the water vapor transmission rate (WVTR) through films was determined using the method described in ASTM E 96-95.[16] Aluminum cups having area of mouth as 46.5 cm^2 were used for mounting of specimens. Desiccant method was used. Anhydrous CaCl$_2$ (Aldrich, AR Grade) was well ground in mortar with pestle to specific size and was dried thoroughly in oven at 125°C for a day and was used as desiccant material. The cups were washed with soap water and distilled water and dried thoroughly in oven. Approximately 40 g of

desiccant was taken in the cup and the samples were cut to exact size to be mounted on test cup. The plasma processed surface was in upper side with the opposite side facing the desiccant material in the cup, samples were held flat preventing wrinkles and crease. The WVTR was measured by weighing cups regularly over a given period of time (24 h). Oxygen transmission rate (OTR) was performed using MOCON OXYTRAN 2/21 instrument.

The morphological studies were carried out using a Philips Scanning Electron Microscope (SEM) (model XL-30). ESCA Thermo VG Scientific MultiLab 2000 was used for chemical analysis.

Curve fitting: Lorentzian nonlinear curve fitting program of ORIGIN-6 software was used.

Principles of ESCA

Surface analysis by ESCA is accomplished by irradiating a sample with monoenergetic soft X-rays and analyzing the energy of the ejected electrons. Monochromatic MgKα (1253.6 eV) or AlKα (1486.7 eV) X-rays are usually employed as source of irradiation. When the sample is irradiated with such monochromatic X-rays, the photoionization of the inner core electrons will occur. The resulting photoelectron will have a kinetic energy given by,

$$E_k = h\nu - E_B - \Phi_s$$

here E_k is the kinetic energy of emergent electron, $h\nu$ is the energy of incident X-rays. E_B is Binding energy of core electron and Φ_s is the spectrometer work function.

Since $h\nu$ and Φ_s are known, and E_k can be measured, E_B i.e., binding energy of core electron can be calculated. Because each element has a unique set of
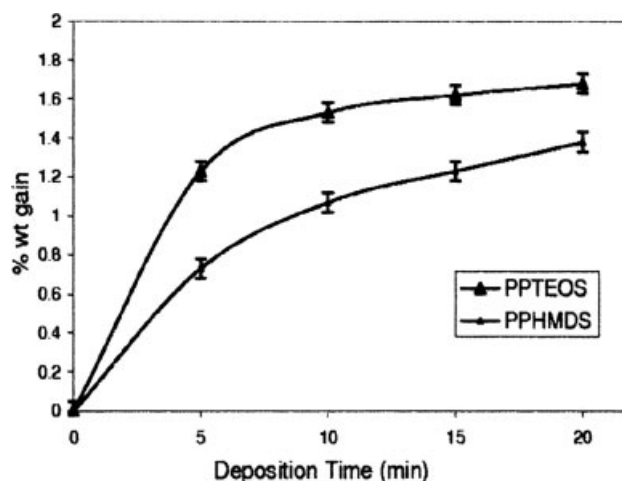

Figure 1   % weight gain study of PPTEOS and PPHMDS.

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TABLE I
Change in Contact and Angle and SE of TEOS Plasma-treated PE

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Time (min)	CA wrt water (W)	CA wrt glycerol (G)	CA wrt formamide (F)	CA wrt ethylene glycol (EG)	γ_s^p polar compo	γ_s^d disperse compo	γ^s (mJ/m^2)
0	88.93	74.47	68.92	55.75	3.49	26.95	30.44
5	75.45	63.06	51.12	46.61	7.933	29.10	37.04
10	66.12	57.02	46.41	39.21	15.33	23.89	39.22
15	55.38	48.62	36.16	32.43	23.67	21.43	45.10
20	48.69	39.63	28.58	10.26	29.12	20.35	49.47

binding energies, ESCA can be used to identify and determine the concentration of the elements on the surface.[17]

RESULTS AND DISCUSSION

Percentage weight change in PE films

The rate of deposition was monitored by measuring the percentage weight change. The rate of deposition on substrate depends on several parameters.[3] Since in this case etching and deposition are simultaneous processes, it is very difficult to determine actual deposition rate on polymer substrate. Figure 1 shows the percentage weight change of plasma polymerized tetraethylorthosilicate (PPTEOS) and plasma polymerized hexamethyldisiloxane (PPHMDS) deposited on PE films for various durations of time. It is clearly seen that percentage weight rises with increase in processing time. Though the etching and deposition occur simultaneously, deposition rate is much faster than that of etching and results in weight gain.

Surface energy measurement of PPTEOS on PE film

Typically plasma treatments are used to add polar functional groups that dramatically increase the surface free energy of polymers. Any change in surface free energy can be easily detected by a change in the contact angle of water with the surface of the modified polymer.[18,19] Plasma polymerization of monomer vapors on polymer substrates have been also used for the modification of properties of substrate polymers.[20]

Plasma polymerization of TEOS was carried out on PE substrate with a special aim to improve barrier properties. It was thought interesting to study surface properties of PPTEOS film, which are equally important. Surface properties have impact on adhesion, printability, seal strength, etc. Table I shows the contact angle with respect to four different liquids was studied to observe the changes in surface energy due to plasma deposition of TEOS on PE substrate. Surface energies of plasma polymers are

mainly dependent on the chemical nature of the monomers used for the plasma polymerization.[21]

The increase in SE is mainly due to the incorporation of polar component (γ_s^p) on to the surface whereas there is decrease in the disperse component (γ_s^p) of the solid surface.

When HMDS was deposited on PE film substrate contact angle was found to decrease drastically, water droplet immediately becomes flat hence we have not measured CA for PPHMDS films. However it can be concluded from the earlier observation that, the surface energy of PPHMDS must be higher. Similar kind of observation was made by Akovali et al.[8]

ATR-FTIR analysis of PPTEOS and PPHMDS on PE film

ATR-FTIR is a convenient method to characterize the bonding modes of SiOx layer. The FTIR spectra of untreated control PE film is shown in Figure 2(a) and its peak assignment is given in Table II. When PPTEOS is deposited onto the PE substrate it can be seen that peaks corresponding CH$_2$ stretching and
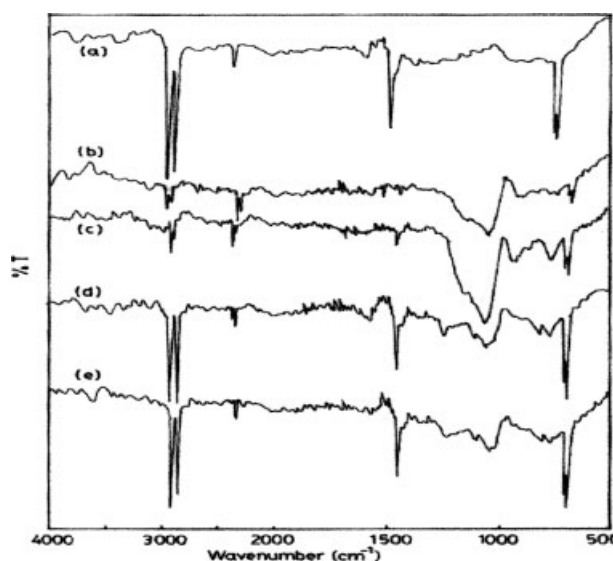
Figure 2 ATR-FTIR spectra of (a) Control PE film. (b) PPTEOS deposited on PE film for 5 min. (c) PPTEOS deposited on PE film for 15 min. (d) PPHMDS deposited on PE film for 5 min. (e) PPHMDS deposited on PE film for 15 min.

TABLE II
Peak Assignment of Control PE

Band (cm^{-1})	Peak assignment
2900	CH stretching
2838	CH_2 stretching
1467	CH_2 bending
1370	CH_2 bending
1290	CH_2 bending
723	CH_2 bending

bending vibration are suppressed as shown in Figure 2(b,c). A strong bands occurs at 1078 cm^{-1} is due to linear (Si—O—Si) and (Si—O—C) groups.[22,23] The peak at 970 cm^{-1} corresponds to C—C. There are also peaks due to the stretching vibration of Si—O groups at 1078 cm^{-1}.[24] The band at 793 cm^{-1} is due to CH_3 rocking at $Si(CH_3)_2$.[9] A peak at 2906 cm^{-1} corresponds to $Si—O—C_2H_5$.[25] It can be also seen that even for higher deposition time peaks corresponding to CH_2 stretching and bending can be seen because CH_2 is present in the structure of PPTEOS.

In case of PPHMDS deposited on PE substrate there is decrease in the intensity of peaks corresponding CH_2 stretching and bending vibration as shown in Figure 2(d,e). As seen from the ATR-FTIR spectra there is a band at 1078 cm^{-1} corresponding to Si—O—Si[24] and a band at 853 and 802 cm^{-1} corresponds to $Si(CH_3)_3$ and $Si(CH_3)_2$.[26] The band at 1258 cm^{-1} corresponds to $Si—CH_3$.[27] A peak at 1371 cm^{-1} corresponds to CH_2 scissor vibration in $Si—CH_2—Si$. A peak at 1451 cm^{-1} corresponds to CH_3 asymmetric bending in $Si(CH_3)_3$. A peak at 2136 cm^{-1} is assigned to Si—H stretching. A peak at 1352 cm^{-1} corresponds to $Si—CH_2—Si$.[9] Peaks at 1421 and 1402 cm^{-1} corresponds to $Si—CH_3$.[25] It is clear from the ATR-FTIR observation that TEOS is getting nicely deposited onto the PE film as com-
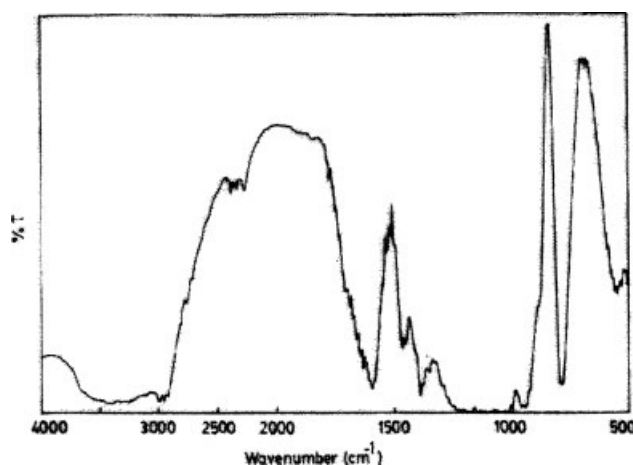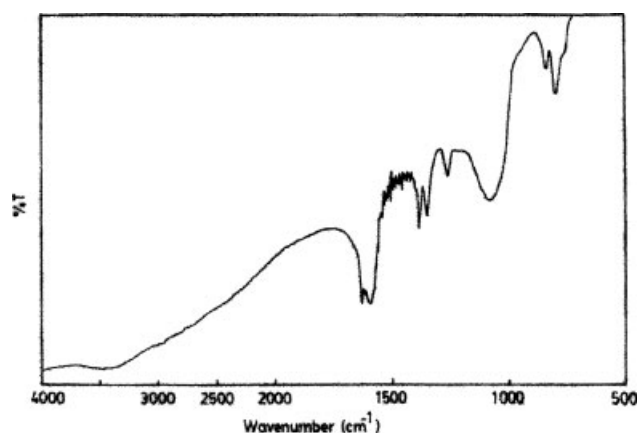

Figure 4 FTIR spectrum of PPHMDS collected from plasma reactor.

pared with HMDS. This is in support with the wt-gain study.

To understand the chemical nature of deposited material in TEOS plasma environment, KBr pellet of powder obtained from walls of reactor was made (2% w/w) and an IR spectrum was recorded. Figure 3 shows FTIR spectra of PPTEOS powder. A broad peak centered at 3400 cm^{-1} because of bonded —OH group is observed. Absorption peaks at 2988, 2915, and 2857 cm^{-1} because of C—H stretching vibration of alkyl group were also observed. Intense peak at 1631.7 cm^{-1} because of nonconjugated C=C stretching was observed. Bands at 1607.6 and 1591 cm^{-1} because of C—O—C was observed. Sharp peaks at 1385.8 and 1350 cm^{-1} were observed for
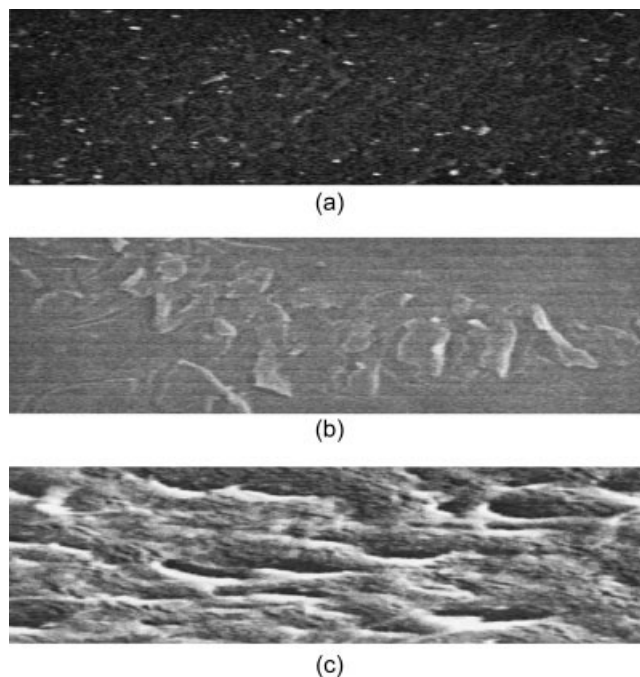

Figure 3 FTIR spectrum of PPTEOS collected from plasma reactor.

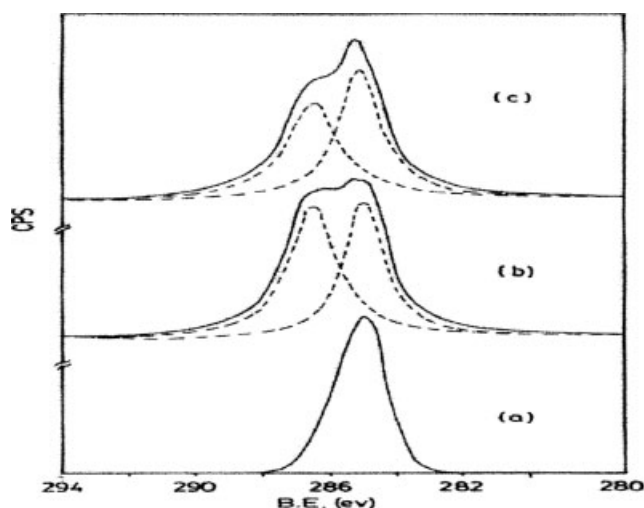**Figure 5** SEM photograph of (a) Control PE (b) PPTEOS deposited on PE film (c) PPHMDS deposited on PE film.

Figure 6 C1s spectra of (a) Control PE film. (b) PPTEOS deposited on PE film for 5 min. (c) PPTEOS deposited on PE film for 15 min.

powder as well as PPTEOS film and are due to CH_2 bending vibration. Very broad absorption band ranging from 1076 to 1093 cm^{-1} was observed due to open chain Si—O—Si stretching vibration. Peaks observed at 1172 and 1163 cm^{-1} are due to Si—O—C stretching vibration. An absorption band observed at 982 cm^{-1} is due to Si—C_2H_5.[28] Weak absorption band because of Si—C group at 795 cm^{-1} was also observed, which is quite broad and definitely due to some contribution of Si—C group.

In case of PPHMDS powder it can be seen from Figure 4 that there is stretching absorption in the region between 1100 and 1000 cm^{-1} corresponding to Si—O—Si, Si—O—C[29] and Si—O[30]. A peak at 1263 cm^{-1} corresponds to Si—CH_3[29] plasma polymerization of organosiloxane). Peaks at 845 and 800 cm^{-1} corresponds to peaks Si—C, Si—CH_3, Si—CH_2.[31] A peak at 763 cm^{-1} corresponds to Si—C. There are also peaks in the region 1636, 1596,1384, and 1354 cm^{-1} corresponding to C=C, C—O—C and CH_2 bending vibration.

Thus IR spectroscopy reveals that plasma polymerization product of TEOS and HMDS mainly gives Si—O—Si structures.

SEM analysis

SEM is a very powerful technique to study the surface morphology of plasma polymerized films.[32,33] The surface of untreated control PE film is shown in Figure 5(a). The PPTEOS and PPHMDS deposited on PE film show snow or flake-like precipitates at the surface as shown in the Figure 5(b,c), respectively. All the photographs have same magnification (15,000×).

ESCA analysis

Figure 6(a) shows C_{1s} spectra of control PE film. It shows only one peak 285 eV, which corresponds to the carbon in CH_2.

When PPTEOS is deposited on PE film, the oxygen which is present in the TEOS structure gets incorporated. Figure 6(b,c) shows C1s spectra of PPTEOS deposited on PE film for 5 and 15 min, respectively. The second peak (287 eV) is comparatively broader which corresponds to C—O and C=O. However when the deposition time is increased from 5 to 15 min, it is observed that the 1st peak which is due to CH_2 has increased because carbon is also present in the similar form of PPTEOS. The second reason is that the first peak (285 eV) has increased because of C—Si which also falls at $\sim$ 284.5 eV.[29]

When PPTEOS was deposited on PE film, it was observed that SiOx layer is formed. Figure 7(a,b) shows the Si_{2p} spectra deposited on PE film for 5 and 15 min. There are two peaks—the first peak which is at 100.7 eV corresponds to Si—C and the second one corresponds to Si—O which is at 102 eV.[29] In the discussion of C_{1s} spectra we have mentioned that the increase in the first peak is due to the carbon which is attached to Silicon and is confirmed from the Si_{2p} spectra (1st peak Si—C at 100.7 eV).

From the atomic concentration Table III it is clear that oxygen is getting incorporated in the form of SiOx layer and is also evident from Si—O (102 eV).

Figure 8(b,c) shows the C1s spectra of PPHMDS deposited on PE film for 5 and 15 min which consist of two peaks one at 285 eV due to C—Si and another at 287 eV due to C—O and C=O.[7] However when the deposition time was increased from 5 to 15 min there is increase in the 1st peak because of C—Si kind of structure is formed on to the surface. This peak is due to the contribution of both CH_3 and
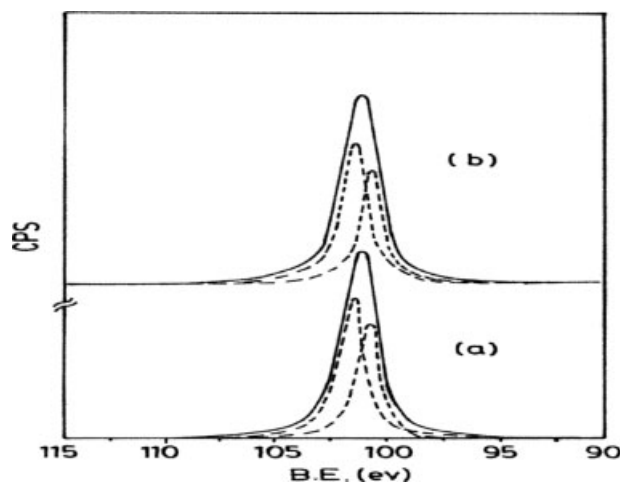

Figure 7 Si_{2p} spectra of (a) PPTEOS deposited on PE film for 5 min. (b) PPTEOS deposited on PE film for 15 min.

TABLE III
Percentage Atomic Concentrations of Various Elements in the PE Sample Subject to Plasma Polymerized Processed PE Film

Sample name	C1s	O1s	Si$_2$p
PE cont	100	–	–
PPTEOS 5 min	40.13	39.44	20.42
PPTEOS 15 min	44.40	33.82	21.78
PPHMDS 5 min	27.41	48.13	26.47
PPHMDS 15 min	24.58	48.56	26.91

C—Si. The Si$_{2p}$ spectra as in Figure 9(a,b) shows 2 peaks—one at 100.7 eV due to Si—C and another at 102 eV due to Si—O.[29] Here also as in the case of Si$_{2p}$ spectra of PPTEOS there is increase in the first peak due to Si—C.

A PE structure consist of no oxygen atoms hence there is no presence of oxygen, which is also confirmed from the ATR-FTIR and ESCA results, but the monomers used i.e., TEOS and HMDS contains oxygen in their structure, hence when plasma polymerization of HMDS and TEOS occurs several oxygen containing moieties are formed such as Si—O, Si—O—Si, and Si—O—C. The percentage of oxygen and Silicone has increased because of plasma processing and is shown in Table III.

ATR-FTIR spectra, which shows the presence of groups like Si—O and Si—C, is in good agreement with ESCA results.

WVTR and oxygen permeability through PPTEOS film and PPHMDS deposited on PE film

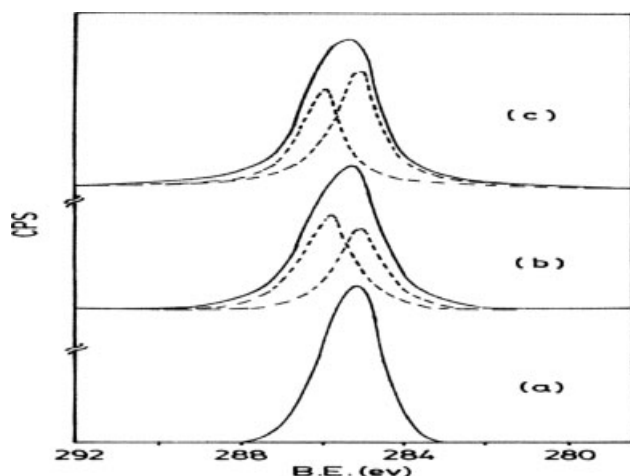Inagaki et al.[15,34] have showed improvement in oxygen barrier property when tetramethylorthosilicate

Figure 8 C1s spectra of (a) Control PE film (b) PPHMDS deposited on PE film for 5 min. (c) PPHMDS deposited on PE film for 15 min.
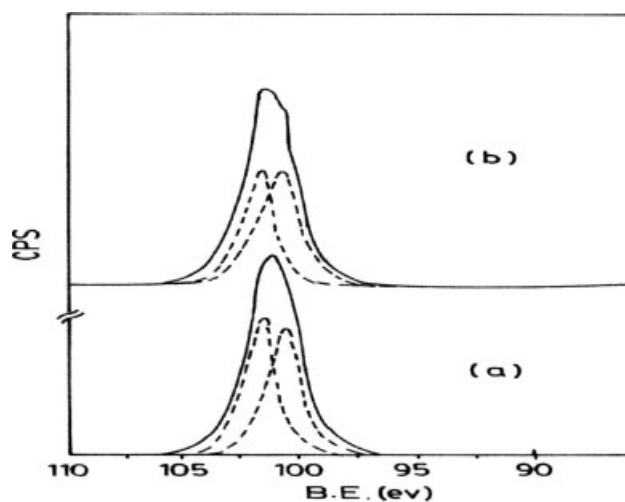

Figure 9 Si$_{2p}$ spectra of (a) PPHMDS deposited on PE film for 5 min. (b) PPHMDS deposited on PE film for 15 min.

(TMOS) was plasma polymerized and deposited on PET and PP film. In the present study TEOS and HMDS was deposited on PE film to enhance barrier properties. Hence, it was thought interesting to study WVTR and O$_2$ permeability of modified PE films. PPTEOS and PPHMDS deposited on PE substrate resulted in decrease in permeation rates of water vapors as shown in Figure 10(a). The study of O$_2$ permeability is very important from packaging point of view. Therefore OTR was studied using MOCON OXYTRAN 2/21 instrument. The working pressure was 23°C and 1 atm pressure. An average of three samples is given in Figure 10(b). It shows decline in permeation characteristics for both the materials however the decrease in OTR in case of PPTEOS is more in comparison with PPHMDS.

The permeability results can be easily explained as due to the resistance offered by the top layer of the PPTEOS and PPHMDS, which is highly crosslinked and pinhole free. Thus this film acts as a two-layered film reducing the permeability of water vapor. Since in the case of PPTEOS, SiOx layer is nicely deposited as compared with PPHMDS as evident from IR, ESCA, and SEM study, hence there is more decrease of WVTR and OTR in case of PPTEOS deposited on PE film. The another reason could be the rate of deposition/polymerization of PPTEOS is more as from the wt-gain study it is evident that PPTEOS wt-gain is more as compared with PPHMDS (Fig. 1). Therefore such modified films can find applications in packaging.

CONCLUSIONS

Plasma polymerization of TEOS and HMDS was carried out on PE films. The barrier properties, surface
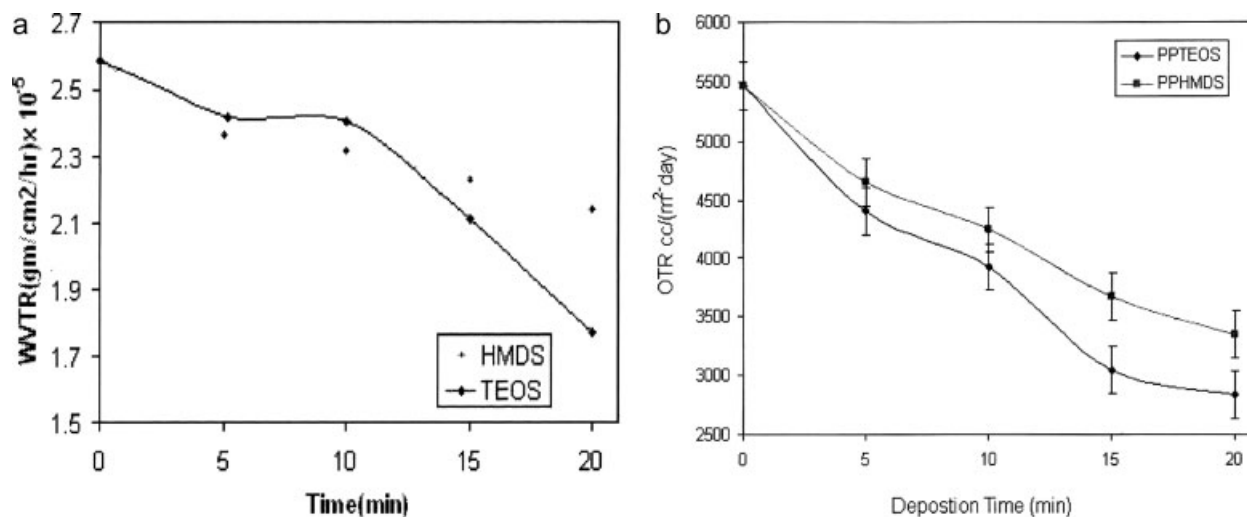
Figure 10 (a) WVTR study of PPTEOS and PPHMDS deposited on PE film (b) OTR study of PPTEOS and PPHMDS deposited on PE film.

properties and chemical composition of the plasma polymerized films was studied. The results are summarized as follows:

1. Plasma polymerization of siloxane containing monomers shows that the monomers containing different function groups possess different reactivity's during plasma polymerization. In plasma polymerization monomer molecules undergo partially selective chemical bond breaking and recombination process to form macromolecules.
2. The monomers of TEOS exhibit high plasma polymerization rates as compared with that of HMDS which is evident from the results obtained from Percentage Weight Gain, WVTR, and OTR study.
3. From the surface energy results it can be seen that plasma polymerization of TEOS and HMDS create hydrophilic and wettable surface as compared with control PE film.
4. The WVTR and OTR results show that plasma polymerization of TEOS and HMDS gives uniform pin-hole free film. The deposition of monomers of these films can be seen from the SEM. With PPTEOS giving much less permeability to water vapor as compared to PPHMDS.
5. Plasma polymerization of TEOS and HMDS yield several silicon related groups like Si—O, Si—C, Si—O—Si at its surface. The XPS and ATR-FTIR of plasma-polymerized film was in good agreement to our observation. Also from the ATR-FTIR spectra it can be seen that monomers of TEOS are nicely deposited on to PE film as compared with HMDS.

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