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Polyethylene Maleate Copolyesters as Coating Materials for Piezoelectric Quartz Crystal-based Chemical Sensors

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ABSTRACT

Polyethylene maleate (PEM) was synthesised by direct polycondensation of maleic anhydride and ethylene glycol in toluene under reflux usingp-toluene sulphonic acid as a catalyst. Structure of PEM was further modified by varying nature of diols and acid components, chain length of glycols, incorporation of aromatic and fluorine groups in the chain. Formation of PEM was monitored by gas chromatography. The parameters like degree of polymerisation, number average molecular weight (Mn), and weight average molecular weight (Mw) were calculated from the extent of reaction and stoichiometric ratio of the reactants. The number average molecular weight was also determined using Knauer vapour pressure osmometer. Cohesive energy, volume, and solubility parameters of PEM-based copolyesters were calculated by group contribution method. These PEM-based copolyesters' have been evaluated as sorbent-coating materials for the detection of organo phosphorus(OP)compo using dimethyl-methylphosphonate (DMMP) as model compound and piezoelectric crystal detector. PEM-bisphenol A is found to be the most sensitive and potential coating material for the detection of OP compounds using piezoelectric crystal detector. Potential PEM-based copolyesters have been characterised by viscosity, infrared spectroscopy, NMR spectroscopy, Mn and thermal stability.

Keywords: Sorbent-coating material, piezoelectrical crystal detector, surface acoustic wave device, organophosphorus compounds, dimethylmethylphosphonate, coating materials, coating material selectivity, coating materials sensitivity, coating materials reversibility, DMMP, polyethylene maleate, PEM, copolyesters, chemical sensors

1. INTRODUCTION

The application of a sorbent thin film to a suitable electronic device represents a general approach to the development of a chemical sensor. These sorbent materials specifically and selectively sorb the vapours of interest and result into decreased frequency of an oscillating piezocrystal.This variation of frequency (ΔF) of a piezocrystal is related to the mass (Msing) of coating materials deposited on the crystal surface area *(A)* in cm^2 and basic frequency (F) of the piezocrystal can be calculated from Sauerbrey's equation' as:

$$AF = -2.3 \times 10^6 F^2 (Ms/A)$$

For detection of any target vapour, it is important to select a coating material which can interact with it selectively, has sufficient sensitivity, has fast speed of response, and reversibly. In the initial stages, stationary-phase materials in gas chromatography, inorganic metal salts and complexes*

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were used as coating materials for the detection of toxic organophosphorus (OP) compounds. However, these coatings were found to be unstable. Polymers were preferred as coating materials due to their better stability and sorption properties. Polymers offer a number of advantages as sorbent-coating materials for the detection of organic compounds. Diffusion of adsorbed vapours within the polymers is quite rapid provided that the glass transition temperature (T_g) is below the operating temperature of the sensors. Polymers suitable as sorbent-coating materials for piezoelectric quartz crystal detector and capable of interacting with the target vapour, have not been reported in the literature. However, polymers like polyethylene maleate (PEM), flouropolyol, hexaflouro-2-propanol (HFIP) containing polymers based on polystyrene and polyisoprene backbones, polysiloxane with pendent HFIP groups (SXFA), and polymers incorporating hexaflouro-bisphenol-A between polydimethylsiloxane (BSP3) and (BSP6), have been reported as coating materials using surface acoustic wave (SAW)sensors[3-6]. PEM is a well-known and potential coating material useful for the detection of OP compounds using surface accoustic wave (SAW) sensors*. To search for coating materials for the detection of OP compounds, PEM was tried as sorbent-coating material for piezocrystal detector. Structure of PEM was also modified by incorporating various groups to increase its selectivity and sensitivity towards adsorption of OP compounds.

Further, no systematic investigations using PEM-based coatings on piezoelectric crystal detector have yet been reported. This paper reports the synthesis, characterisation, and evaluation of PEM-based copolyesters as sorbent-coating materials for the detection of OP compounds using DMMP as model OP compound and piezoelectric crystal detector.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

All the chemicals required for the present study, ie, maleic anhydride (MAn), bisphenol A (BA), hexafluorobisphenol A (HFBA), p-toluenesulphonic acid, isophthalic acid (IPA), terephthalic acid (TPA) (all from Fluka) and ethylene glycol, polyethylene glycol 400 (PEG-400), PEG-4000(Merck), 1,2 and 1,3 propanediol and, 1,2 and 2,3 butane diol (all from Aldrich) were procured from trade and used as received.

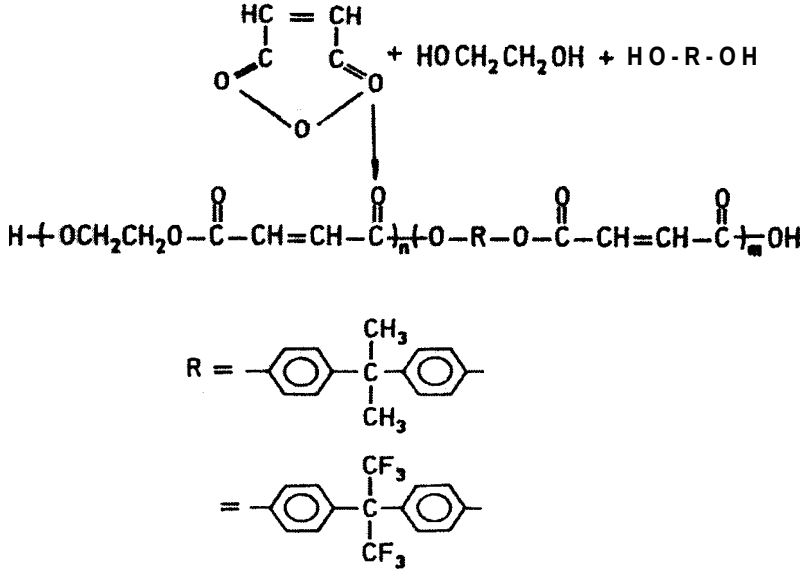

Figure 1. Synthesis of polyethylene maleate-based polyesters

2.2 Synthesis

Polyethylene maleate was synthesised[7] by an acid catalysed (*p*-toluene sulphonic acid 1 .O x 10^{-3} mol/mol of glycol) polyesterification of maleic anhydride and ethylene glycol using Dean-Stark apparatus (Fig. 1). Stoichiometric amounts of maleic anhydride and glycol (1 .O: 1.1) were refluxed in toluene under nitrogen atmosphere. Water formed during the reaction was removed as an azeotrope with toluene. Modified copolyesters were also prepared keeping the glycol: acid ratio 1.1: 1 .O same. Solvent was stripped off and polymers were isolated and dried. Formation of PEM was monitered by withdrawing aliquots from the reaction mixtures at different intervals of time and analysing the amount of residual monomers by chemito gas chromatograph equipped with BP- 10 column and flame-ionisation detector. The column (30 m x 0.5 mm i.d.) was temperature programmed from 60 °C to 125 °C @ 4 °C per minute.

2.3 Characterisation

Polyethylene maleate-based polyesters were characterised[8] using the following techniques:

(i) *Viscosity:* Ubbelhode viscometer was used to determine the viscosity of 0.5 per cent solution of polyesters in acetonitrile at 30 "C.

(ii) *Number average molecular weight* (Mn): Vapour pressure osmometer (Knauer, Germany) was employed for determining Mn of polyesters.

(iii) *Acid number:* Each polyesters was dissolved in toluene and reacted with alcoholic sodium hydroxide using phenolphthalein as indicator. Acid number was expressed as mg of *KOH*/g of polymer.

(iv) *Thermal stability:* The polyester specimen were heated at 150 °C for 4 h and per cent weight loss was determined gravimetrically. T_g of PEM was determined by DSC-2920 (TA instruments, USA).

(v) *Infrared spectroscopy:* IR spectra of polyesters were recorded in *KBr* using Nicolet FTIR 410 spectrophotometer in the range 400-4000 cm-'

(vi) *NMR:* Polyesters were also characterised by ^1H NMR (90 MH Joel) in $CdCl_3$ using trimethyl silane as an internal standard.

2.4 Crystal Coating

The piezocrystals used were AT cut, spherical quartz crystals of 7 mm dia and 0.2 mm thickness with a basic resonant frequency of 10 MHz and provided with circular gold electrodes on both the sides. The crystal was coated by each polymer solution in acetone (10 mg /1 0 ml) using a microsyringe on both surfaces of quartz crystal by solvent evaporation technique. The amount of coating was about 7.0 µg (after drying at 100 °C/2 h and 1 .O mm *Hg* of vacuum), which was estimated from the frequency change after coating according to Sauerbrey's equation'.

2.5 Apparatus

Figure 2 shows the experimental setup of a PZ quartz crystal detection system. The PZ crystal was housed in a glass cell. The input of this cell was connected to the vapour generator (Graseby Analytical, UK) and output was connected to exhaust. The vapour generator provided vapours of DMMP of a fixed concentration. The vapour generator was calibrated using instrument AP2C (Proengin-E tat Francais) followed by gas chromatography.

The cell housing the PZ crystal was fitted at the outlet nozzle of the vapour generator and purged with zero air for about 5 min. The reading of the crystal oscillator frequency in the frequency counter was noted. This frequency was considered as the baseline frequency. After this, the crystal was exposed to DMMP vapours (saturated concentration and concentration of 0.45 mg/m³). The frequency of the crystal oscillator started reducing and became stabilised as denoted by the frequency counter. The difference of this frequency from the baseline frequency was the frequency drift (AF in Hz), which was characteristic of sensing signal of the piezoelectric crystal detector circuitory.

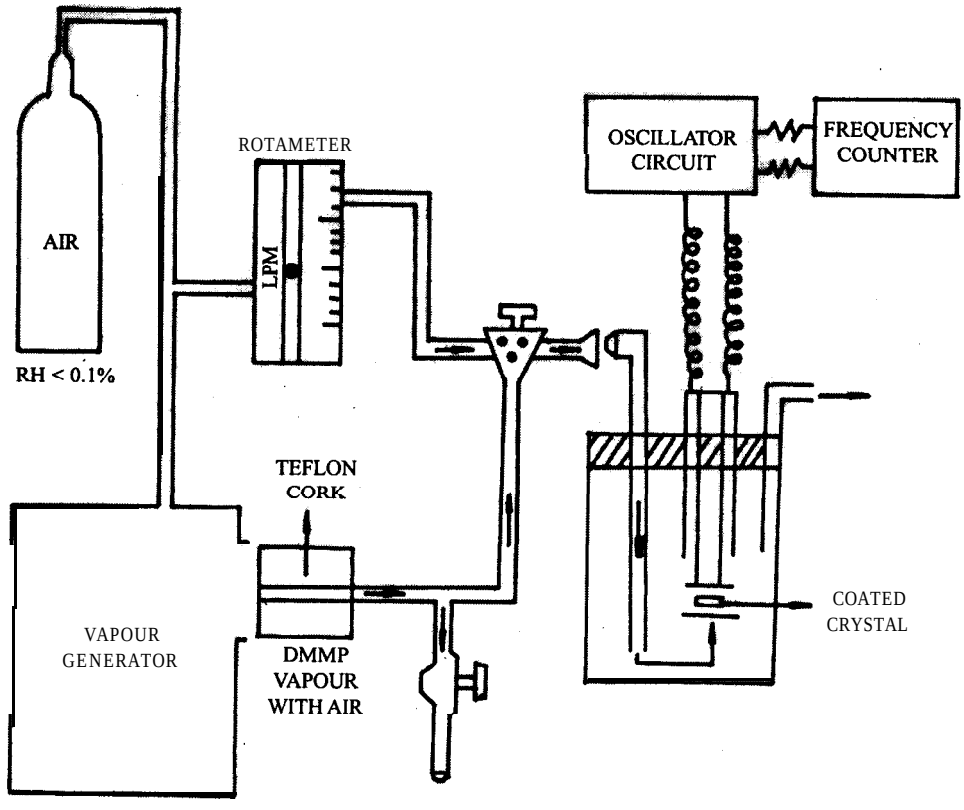

Figure 2. Test assembly for quartz, piezoelectric crystal detector

3. RESULTS & DISCUSSION

The PEM was synthesised as per the reported method of Snow and Wohltjen[7] by acid catalysed polycondensation of maleic anhydride and ethylene glycol in toluene using Dean-Stark apparatus as per the scheme given in Fig. 1. Formation of PEM was monitered by gas chromatogrphy (Fig. 3). It was observed that 95 per cent ethylene glycol was consumed in 24 h, and hence, extent of reaction (p) is 0.95 as given in Table 1.

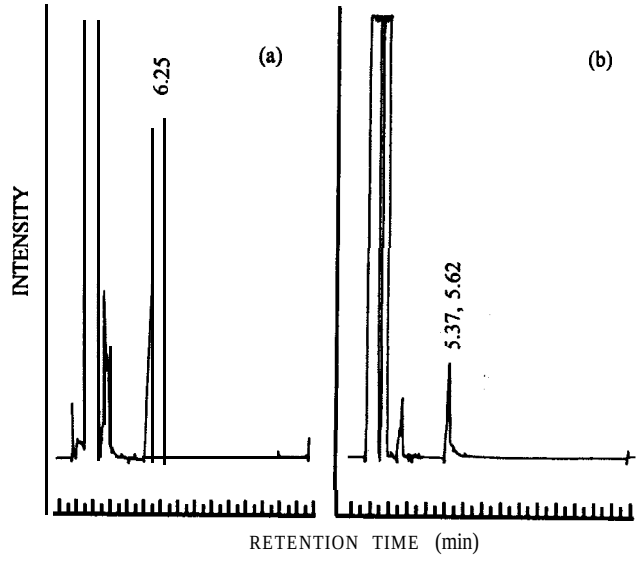

Figure 3. Gas chromatograms showing the presence of ethylene glycol in reaction mixture at (a) zero time; RT 6.25 min (b) after 24 h; RT 5.37, 5.62 min.

By substituting p = 0.95 in standard equation[9], the number average molecular weight (Mn) was

Table 1. Determination of the extent of reaction by gas chromatography

Time (h)	Residual monomer (mol)	Residual monomers (%)	Extent of reaction (p)
0	0.1000	100.0	---
1	0.0200	20.0	0.80
6	0.0140	14.0	0.86
16	0.0072	7.2	0.93
24	0.0050	5.0	0.95

Table 2. Calculated values of Dp, Mn, Mw, and polydispersity in PEM

Parameter	Value	Defining equation
Stoichiometric ratio (r)	0.909	$r = n_a / n_b$ (n_a n_b: No.of moles of diacid & diol)
Extent of reaction	0.950	p_n= (n-l) / (n+l), n number of repeat units (39.0)
Degree of polymerisation (Dp)	10.500	$Dp = (r+1) / (r+1-2rp)$
Polydispersity (S)	1.950	$S = Mw / Mn$
Number avearage Mol. Wt. (Mn)	3160	Mn = Mo / (l-p), Mo Mol. Wt.of repeat unit
Weight average Mol. Wt. (Mw)	6162	$Mw = Mo(1+p) / (l-p)$

calculated to be 3 160 (Table 2), which is in conformity with the value obtained with vapour pressure osmometer. This polymer was coated over piezocrystal in the form of solution using a microsyringe. Piezoquartz crystal coated with PEM was exposed to saturated DMMP vapours and shift in frequency of the crystal detector was noted. PEM has been found to be suitable as a sorbent-coating material for piezoquartz crystal as indicated by its interaction with DMMP (OP compound), resulting in shift of frequency of the crystal detector. Frequency shift value, **AF** =1564 was not high or significant when PEM was used as a sorbent-coating material. However, PEM has advantages of long term stability (> 6 months) in comparison to inorganic complexes[2]. PEM is a well-known and potential coating material useful for the detection of OP compounds.

To increase the sensitivity and selectivity of PEM towards OP compounds, structure of PEM was modified. Prior to this, analyte-polymer interaction was theoretically predicted. The relative importance of structural features in enhancing the crystal detector sensitivity is difficult to quantify. Nevertheless, the analyte-polymer interaction can be rationalised, to some extent, by modelling the dissolution of solute (DMMP) vapours in a solvent coating. Since all polymers were employed above their glass transition temperatures[19], these can be considered as solvents. The cohesive energy (E_{coh}), volume (V), and solubility parameter (6) of the polymer influence the dissolution process. These parameters were theoretically calculated from the group contribution method[10]. In spite of E_{coh}(63-161 kJ/mol), the increased volume of the derivatives of PEM (Table 3) may be responsible for facilitating the uptake of DMMP. Furthermore, lesser the difference in solubility (6) of DMMP and polymers, higher is its sorption. The solubility of DMMP and PEM was calculated to be 15.0 $J^{1/2}/cm^{3/2}$ and 28.5 $J^{1/2}/cm^{3/2}$ respectively. Upon copolymerisation, the solubility of PEM is lowered, and hence, the sorption of DMMP is favoured. Thus, it was expected that modified copolyesters have better interaction with DMMP (analyte vapour) and lead to increase sensitivity of the peizoquartz crystal detector. Therefore, the structure of PEM was modified to achieve the higher sensitivity of the piezo detector.

Table 3. Calculated cohesive energy (E_{coh}), volume (V), and solubility parameter (6) for PEM and its derivatives

Polymer	Modifying unit	E_{coh} (kJ/mol)	V (CC/mol)	δ ($J^{1/2}/cm^{3/2}$)
PEM	Control	63.29	77.7	28.50
PEM-BPA	Bisphenol A	75.07	103.1	26.98
PEM-6FBPA	6FBPA	74.91	115.3	25.49
PEM-P	2-Propanol	71.43	109.6	25.52
PEM-PEG	PEG-100	161.31	349.2	21.49

Table 4. Normalised DMMP vapours responses of PEM-based polymers using piezoquartz crystal detector

Polymer	Composition mol	AF, Hz /3000 Hz at DMMP concentration	
		Saturated concentration	0.45 mg/m³
PEM-1	MA+EG	1564.0	12.0
PPM	MA+1,3 Propanediol	880.0	--
PPM	MA+1,2 Propanediol	1041.0	--
PBM	MA+1,2 Butanediol	1445.0	--
PBM	MA+2,3 Butanediol	1590.0	--
PEM-2	MA+DEG	1906.0	--
PEM3	MA+EG+PEG-400	1955.0	--
PEM-4	MA+PEG-400	2699.0	14.0
PEM-BA	MA+EG+B-A (0.2)	3474.0	19.0
PEM-BA	MA+EG+B-A (0.5 mol)	2534.0	--
PEM-IPA	MA+EG+IPA (0.2 mol)	1229.0	--
PEM-IPA	MA+EG+IPA (0.4 mol)	2739.0	--
PEG-TPA	MA+EG+TPA (0.4 mol)	2698.0	
PEG-HBA	MA+EG+HBA (0.2 mol)	3180.0	16.0
PEG-HBA	MA+EG+HBA (0.4 mol)	2375.0	--

Standard deviation = ± 0.5 Hz at DMMP concentration 0.45 mg/m³

Three series of polyethylene maleate-based polyesters were synthesised by varying (i) nature of diols and acid components, (ii) chain length in glycols and their polarity, (iii) incorporation of aromatic group in the chain. These modified polyesters synthesised are given in Table 4. Each of these modified polyesters was coated over the piezocrystal in the form of a solution. The coated crystal was exposed to DMMP vapours at saturated concentration and shift in frequency was noted. It was observed that. response of the detector was better when these modified polyesters were used as sorbent-coating materials than inorganic copper complexes[2]. Further, these modified polyesters give an alarm at saturated concentrations of DMMP. It was also observed that by increasing chain length in glycols (by incorporating diethylene glycol, PEG-400 along with ethylene glycol), the response and sensitivity of the piezoelectric quartz crystal were increased (Table 4).

Table 5. Characteristics of polyethylene maleate-based polymers

Property	Polymers		
	PEM	PEM-BA	PEM-HBA
Colour	Colourless (transparent)	Light brown	Pale yellow
State	Viscous gum	Viscous gum	Viscous gum
Relative viscosity	1.00	0.08	0.07
Mn	2940	2580	1850
Acid number (mg/g)	22	39	44
Weight loss (%)	10.3	12.0	13.7
Response time (s)	40	30	40
Recovery time (min)	3.0	2.0	2.0
Stability period (month)	6	6	6

Incorporation of aromatic moiety in PEM (bisphenol A, isophathalic acid, terephthalic acid, etc) also increases the sensitivity of the crystal detector due to increase in hydrophobicity of these polymers.The potential sorbent-coating materials, as given in Table 4, were also tested for DMMP concentration of 0.45 mg/m³. PEM containing 0.2 mol bisphenol **A** was found to be the most selective, sensitive, and potential coating material giving AF value of 3474 Hz at saturated concentration and 19 Hz/3000 Hz at DMMP concentration of 0.45 mg/m³. Incorporation of fluorine group along with aromatic moiety by incorporating 0.2 mol of hexafluorobisphenol also gives good detector response (AF = 16 Hz). This leads to the conclusion that incorporation of aromatic moiety and polar fluorine group in PEM also increases the sensitivity of the crystal detector. PEM interacts with DMMP by hydrogen bonding. Incorporation of highly electronegative fluorine atom in PEM structure increases its interaction with DMMP by dipole-dipole interaction mechanism³. Again, these polymers, ie, PEM containing 0.2 mol of bisphenol A and hexafluorobisphenol A were further tested for response time, recovery time, and stability of the detector. It was observed that response time of 30-45 s and recovery time of 2-3 min was observed for these polymers. Similarly, when these polymers were used as sorbent-coating materials, these polymeric coatings were able to detect DMMP for more than 6 months, indicating that these polymers are more stable than any other sorbent-coating material reported so far. These polymers, which are potential coating materials, have been characterised for further studies.

3.1 Characterisation

The PEM is transparent and colourless liquid at room temperature. It is soluble in organic solvents like toluene, acetonitrile, tetrahydrofuran (THF) and *N*,*N*-dimethylformamide (DMF), etc. Relative viscosity of PEM was found to be low (< 0.1) at 30 "C, indicating oligomeric nature of this polymer. This is further supported by Mn = 2940 and acid number of 22 mg of *KOH*/g of PEM. PEM containing 0.2 mol bisphenol A is light brown liquid, whereas PEM having hexafluorobisphenol A is light yellow liquid. Their relative viscosities are low (0.07–0.08), indicating the oligomeric nature of these polymers.

Further Mn and acid number values are found to be 2580 mg and 39 mg of *KOH*/g respectively for PEM-BA. Similarly the values are Mn=1850 and acid number 44 mg of *KOH*/g for PEM-HBA. PEM has T_g value of 8 "C.

The FTIR spectra of PEM indicate strong peak of 1731 cm^{-1} due to carbonyl group, indicating the formation of ester. Peaks at 1642 cm-' indicate the presence of disubstituted *C=C* stretching (Fig. 4). Peaks at 3499 cm^{-1} and 2957 cm-' may be due to the presence of *-OH* and *C-H* stretching. The FTIR spectra of PEM containing bisphenol A also shows a strong peak at 1728 cm-' due to *C=O* group. A strong and broad band at 3433 cm^{-1} was due to the presence of *-OH* group in PEM. Peak at 2965 cm^{-1} may be due to *C-H* stretching. Aromatic *C=C* stretching bands at 1513 cm-' and 1639 cm^{-1} were also observed. The presence of *C-F* band at 1258 cm^{-1} in hexafluorobisphenol A containing PEM along with peaks at 1732 cm^{-1} due to carbonyl group, peak at 3435 cm^{-1} due to *-OH* and peak at 2963 cm-' due to *C-H* stretching was also observed in the IR spectra of PEM containing HFBA. The FTIR spectra of these polyesters is in conformity of the reported literature*. The NMR chemical shifts (solubility in $CdCl_3$) were observed at 4.4 ($-CH_2$ group) and 6.3 ($=CH-$ moieties) in the ratio 12:6 in PEM (Fig. 5). Aromatic protons in bisphenol A and hexafluoro-bisphenol A containing PEM were appeared at δ values 6.6, 6.8, and δ values 6.9, 7.3, respectively. Thermal stability of these polymers was also determined and weight loss was found to be 10-14 per cent at 150 °C for 4 h.

4. CONCLUSIONS

The PEM containing 0.2 mol of bisphenol A and hexafluorobisphenol A respectively were found to be potential sorbent-coating materials for piezocrystal detector for the detection of OP compounds as indicated by AF values 19 Hz and 16 Hz, respectively using 10 MHz quartz crystal. Though these values look to be numerically low, but AF values will increase many-fold when quartz crystal of higher

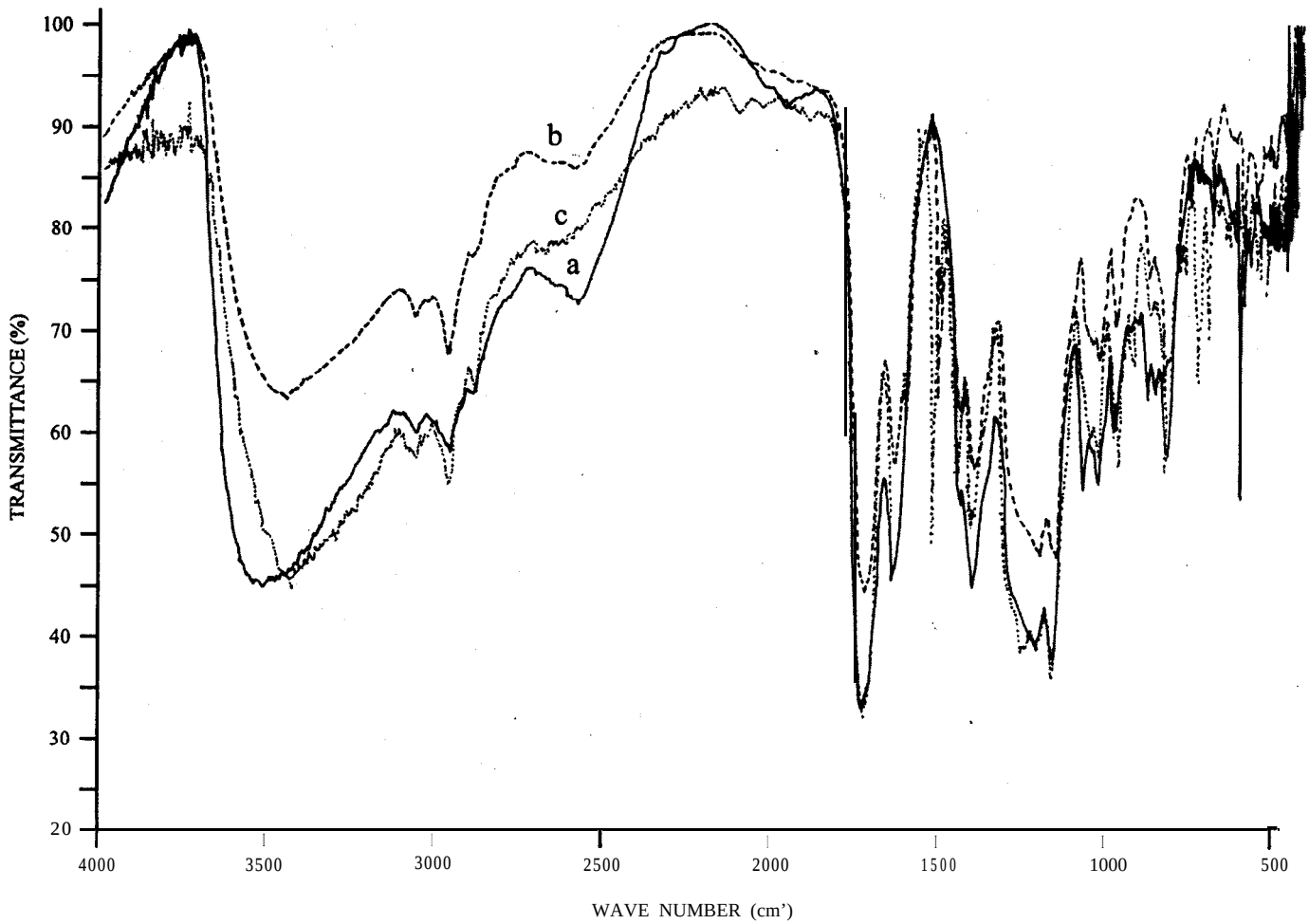

Figure 4. FTIR spectra of (a) PEM, (b) PEM-BA, and (c) PEM-HFBA

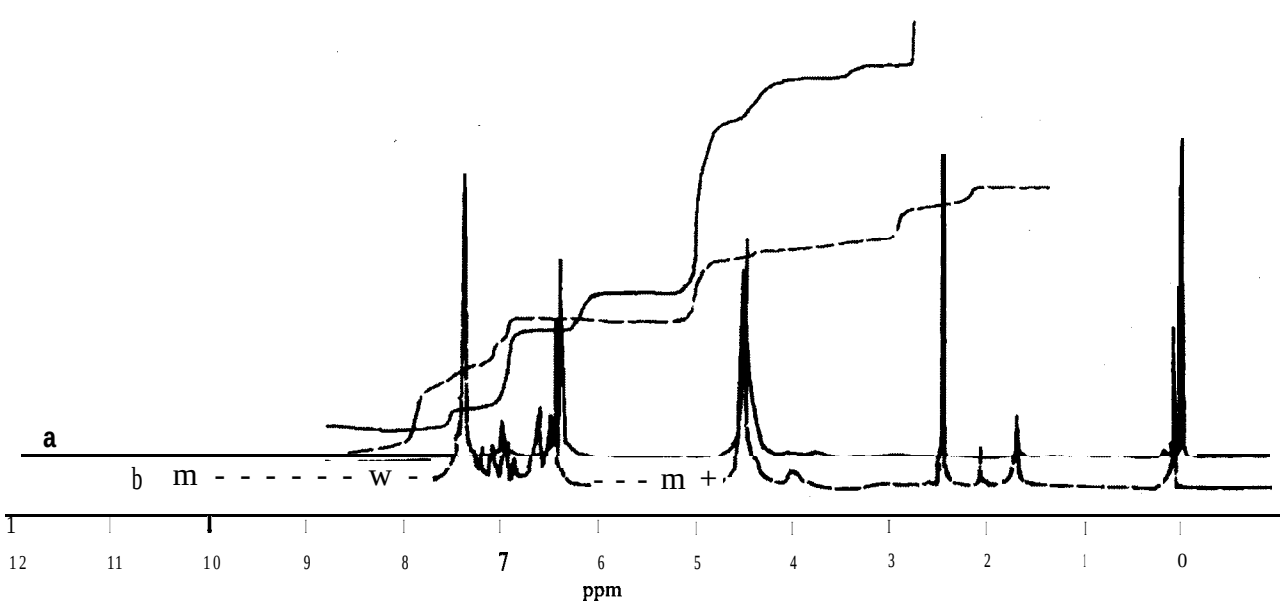

Figure 5. NMR spectra of (a) PEM and (b) PEM-BA

frequency (100 MHz-400 MHz) were utilised for surface acoustic wave (SAW) devices.

Reported AF value[11] for DMMP at concentration 559 µg/l using PEM as coating material with 158 MHz SAW device is 32 Hz, suggesting that the values obtained are superior since these measurements were carried out using 10 MHz quartz crystal. Further, these modified polyesters will be helpful in the development of SAW devices and sensor arrays.

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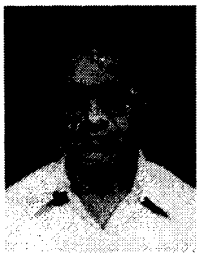

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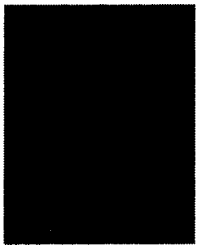

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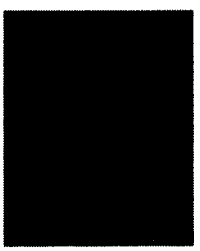

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