

# Hyperbranched Polyesters with Internal and Exo-Presented Hydrogen-Bond Acidic Sensor Groups for Surface Acoustic Wave Sensors

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**ABSTRACT:** Three hyperbranched aromatic polyesters carrying both internal and exo-presented hydrogen-bond acidic phenol, carboxylic acid, or mixed phenol/acetoxo groups were coated onto 500 MHz surface acoustic wave (SAW) sensor platforms, and sensor responses to the nerve agent simulant dimethyl methylphosphonate (DMMP) and the explosives simulant dinitrotoluene (DNT) were studied. All three hyperbranched polyesters gave strong responses to DMMP, and the hyperbranched polyester carrying carboxylic acid groups gave a particularly strong

response. The hyperbranched polyester carrying phenol groups gave the best response to DNT of the three polymers studied. The DMMP and DNT responses of the three hyperbranched polyesters were also compared with hyperbranched SAW sensor polymers carrying exo-presented phenolic sensor groups only, and also with linear SAW sensor polymers carrying phenolic sensor groups. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 107: 1401–1406, 2008

**Key words:** sensors; polyester; hyperbranched; coatings

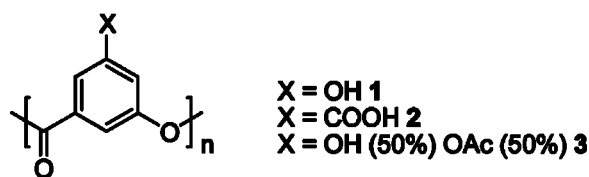
## INTRODUCTION

Gravimetric, optical, chemiresistive, or electrochemical sensor devices operating on an array principle require that each element in the array be coated with a different material, and that this collection of materials span a broad range of solubility interactions, i.e., dispersion, dipole–dipole, and hydrogen-bonding.<sup>1,2</sup> This is usually accomplished using a chemically diverse group of polymers with nonpolar, polarizable, dipolar, hydrogen-bond basic, or hydrogen-bond acidic characteristics.<sup>3–7</sup> Polymers from all of these classes are commercially available, except for the hydrogen-bond acidic class. Hydrogen-bond acidic polymers carrying phenol or fluorinated aliphatic alcohol groups are vital components in surface acoustic wave (SAW) sensor arrays for the detection of hydrogen-bond basic entities such as nerve agents and nitroaromatic explosives in security and defense applications.<sup>1,2,4,8–12</sup> Most SAW polymers used to date have had linear architectures, but hyperbranched architectures have two potential advan-

tages: a higher density of exo-presented sensing groups that may result in higher sensitivity, and a lower density of segmental entanglements that may result in faster vapor diffusion and faster sensor response. Hyperbranched polymers have been used in various electroactive<sup>13–16</sup> sensing and fluorescence<sup>17–19</sup> sensing applications, but have only been reported in four SAW sensor applications. A hyperbranched polycarbosilane carrying both terminal and internal sensor groups was synthesized from the AB<sub>2</sub> monomer *bis*(allyl)-2-naphthylmethylsilane, and hydrogen-bond acidic hexafluoroisopropanol groups were subsequently introduced by the reaction of hexafluoroacetone with the allyl and naphthyl groups.<sup>20,21</sup> This polymer was characterized by FTIR, and it reportedly gave a strong response to the nerve agent simulant dimethyl methylphosphonate (DMMP) when coated onto a SAW sensor. In the second example, dendrimers with halogenated alcohol or phenol end-groups were prepared and used.<sup>22,23</sup> In the third example,<sup>12</sup> hyperbranched SAW polymers carrying terminal hydrogen-bond acidic phenol and fluorinated alcohol sensor groups, but no internal sensor groups, gave good responses to the nerve agent simulant DMMP, and the phenol polymers out-performed the fluorinated alcohol polymers. In the fourth example, a hyperbranched polyester carrying both internal and terminal hydrogen-bond acidic phenol groups, and coated onto an 80 MHz SAW platform, gave

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**Figure 1** Linear repeat unit of hyperbranched polyester carrying both interior and exo-presented hydrogen-bond acidic groups.

good responses to ammonia.<sup>24</sup> In the light of these results, this hyperbranched polyester<sup>24,25</sup> (Fig. 1) was selected for further study with DMMP, and also with the explosives simulant dinitrotoluene (DNT). Its SAW responses to DMMP (Table I) were also compared with hyperbranched polymers carrying terminal phenols only (Fig. 2)<sup>12</sup> and with linear phenol polymers (Fig. 2). Hyperbranched polyesters of this type have already shown promise as sensors that show selective swelling as a function of end group X (Fig. 1) and of analyte vapor.<sup>26,27</sup> Arrays of these materials have also been used to quantify quaternary mixtures of alcohols in water by monitoring differences in sorption and desorption kinetics using reflectometric interference spectroscopy.<sup>27,28</sup>

## EXPERIMENTAL

### Synthesis

Hyperbranched aromatic polyesters were prepared from AB<sub>2</sub> monomers using a melt condensation polymerization technique, and their synthesis and characterization have been described elsewhere.<sup>29–32</sup> These polymerizations generate hyperbranched structures containing dendritic, linear, and terminal units. HB-PE-Phenol **1** was prepared from 3,5-trimethylsilyloxy benzoyl chloride and had a  $T_g$  of 227°C (DSC7, Perkin

Elmer, USA), a molecular mass ( $M_w$ ) of 45,500 and a degree of branching of 0.6. HB-PE-COOH **2** was prepared from 5-acetoxypthalic acid and had a  $T_g$  of 214°C, a molecular mass ( $M_w$ ) of 34,000 and a degree of branching of 0.5. HB-PE-COOH **2** contains a large proportion of COOH groups (10% OH of COOH sensor groups by weight) and only a small proportion of ROH groups resulting from the focal unit (1.5% OH by weight). HB-PE-Phenol OAc **3** was prepared from 3,5-diacetoxy benzoic acid and had a  $T_g$  of 163°C, a molecular mass ( $M_w$ ) of 33,200 and a degree of branching of 0.5. The resulting polymer was partly hydrolyzed. The number of monomer units containing OH groups after hydrolysis was calculated from the <sup>1</sup>H NMR spectrum (DRX 500, Bruker, Germany), and was found to be 22.5 mol %. The  $M_w$  of HB polymers containing OH, COOH, and OH/OAc terminal groups was determined by GPC (Agilent HP 1100, Germany) in DMA using linear PVP (PSS) as a standard.

### Coating

The HB-PE polymers above were coated onto three 500 MHz SAW sensors (Sawtech, TriQunit Semiconductor, Hillsboro, OR) using an airbrush containing 0.05% w/v solution. Polymers **1** and **3** were coated from an acetone solution and polymer **2** was coated from a methanol solution. The thickness of the coatings corresponded to a frequency decrease of 500 kHz measured against a sealed reference SAW mounted on a frequency readout circuit board.

### SAW testing

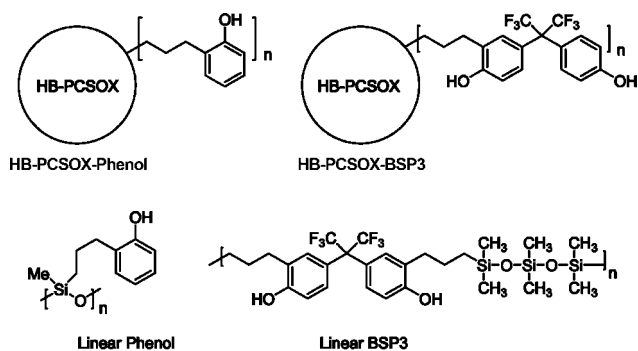
SAW responses to 0.05 ppmv DMMP vapor at 28°C ± 0.3°C and to 0.045 ppmv DNT vapor at 28°C ± 0.3°C were measured using a Femtometrics Indi-

**TABLE I**  
**SAW Sensor Responses, Percent Weight Hydroxyl Contents, and Glass Transition Temperatures for Hydrogen-Bond Acidic Hyperbranched Polyesters and Polycarbosiloxanes, and for Phenolic Linear Polymers Carrying the Same Sensor Groups**

Polymer	DMMP response (Hz)	DNT response (Hz)	OH content (wt %) <sup>a</sup>	$T_g$ (°C) <sup>b</sup>
HB-PE-Phenol <b>1</b>	1759	1160	13	227
HB-PE-COOH <b>2</b>	5292	352	11.5	214
HB-PE-OH-OAc <b>3</b>	2527	461	11	163
HB-PCSOX-Phenol	2740	403	4	–
HB-PCSOX-BSP3	3629	330	5	–
Linear Phenol	3263	323	9	–21
Linear BSP3	5148	717	5	6

<sup>a</sup>Theoretical values of percent weight OH content for the HB-PE polymers were calculated from the theoretical average where each repeat unit carries one functional group (i.e., 50% of the repeat units carry 1 end group, 25% carry 2 end groups, and 25% carry no end group). For HB-PE-COOH **2**, 10 wt % is contributed by OH groups that are part of COOH groups and 1.5 wt % is contributed by focal OH groups. Percent weight OH content for the HB-PCSOX polymers was calculated using quantitative NMR data obtained for the terminal groups in the hyperbranched polymer synthetic precursors.<sup>12</sup> Percent weight OH content for the linear polymers was calculated directly from the repeat unit chemical structure.

<sup>b</sup>Literature  $T_g$  values for linear phenol<sup>33</sup> and BSP3<sup>34</sup> are quoted.



**Figure 2** Hyperbranched polycarbosiloxanes (HB-PCSOX) carrying exo-presented phenolic sensor groups and their linear polymer analogs.

vidual Vapor Detector (IVD) containing a beat frequency reference SAW and an uncoated thermal reference SAW, and the IVD was connected to a vapor generator (VICI Dynacalibrator Model 340) containing a DMMP vapor tube (custom-made) or a dinitrotoluene vapor tube (VICI part number 107-143-7464-C-PE90). The SAW response is quoted and displayed as a positive difference frequency relative to the reference SAW. A more detailed description of the procedures and apparatus associated with SAW cleaning and testing has been published elsewhere.<sup>12</sup>

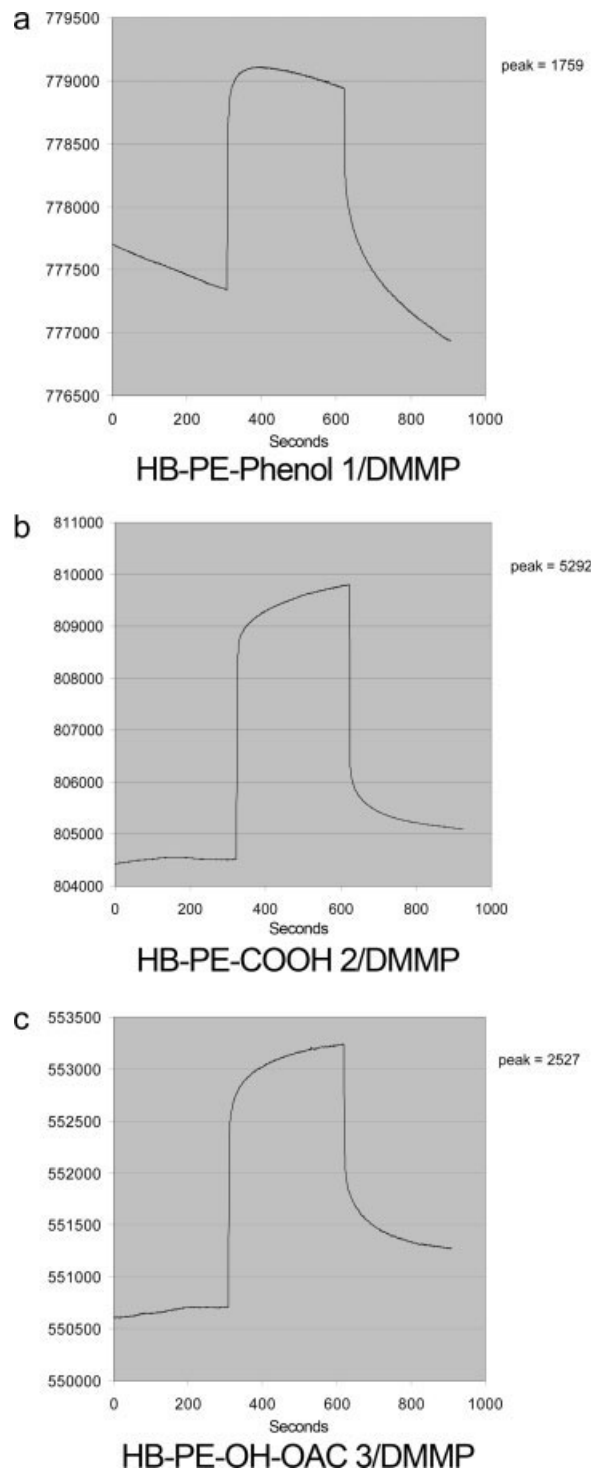
## RESULTS AND DISCUSSION

### SAW responses to DMMP and DNT

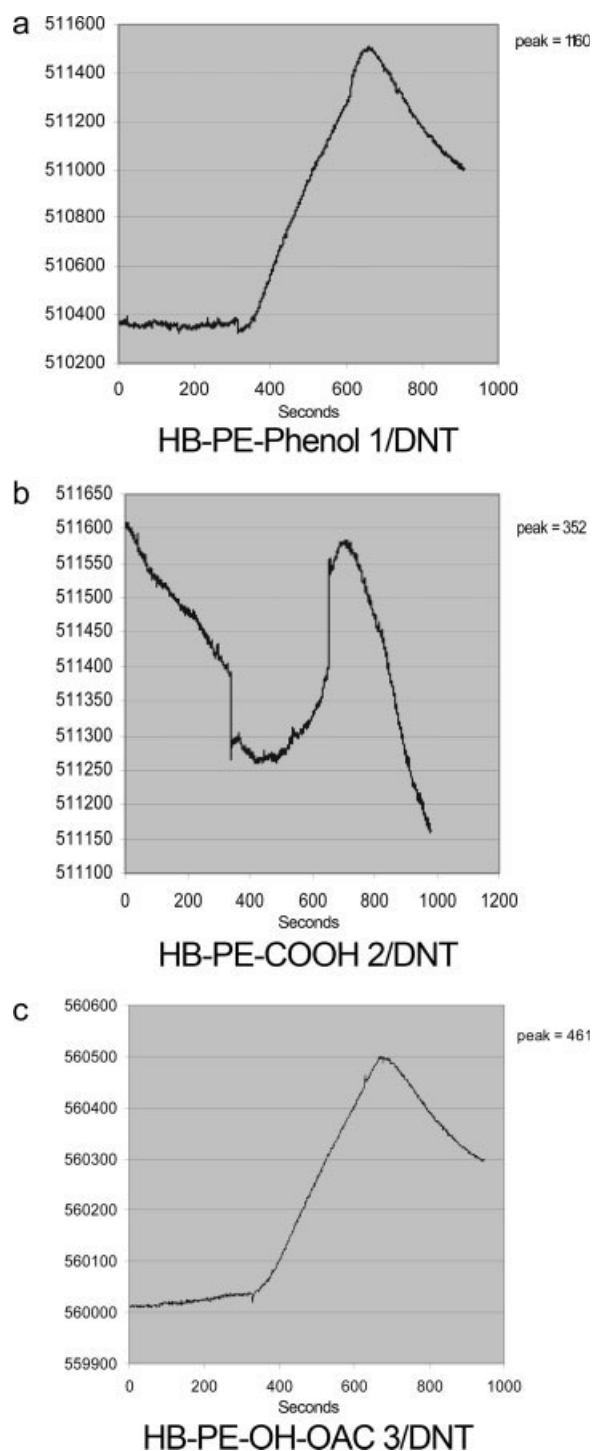
The DMMP SAW response plots for the three HB-PE polymers are shown in Figure 3, and the DNT SAW response plots for the three HB-PE polymers are shown in Figure 4. The SAWs were exposed to 5 min (300 s) of purified air (corresponding to the left-hand side of the plots), followed by 5 min vapor (corresponding to elevated part of the plots), followed by 5 min of purified air (corresponding to the right-hand side of the plots). The height of the peak represents the positive frequency difference between the coated SAW and the reference SAW. The DMMP plots in Figure 3 show quick responses, quick returns to baseline, and good recovery after vapor exposure. The DNT plots in Figure 4 show slower responses and slower returns to baseline. SAW responses of this type are common for low vapor pressure analytes such as nitroaromatics, and have been discussed elsewhere.<sup>33</sup>

The DMMP and DNT SAW responses for HB-PE polymers carrying both terminal and internal sensor groups are shown in Table I. The DMMP and DNT responses of two HB-PCSOX polymers carrying terminal groups only, and two linear polymers are also shown in Table I. All the DMMP and DNT SAW responses in Table I were measured during this study

with the exception of the four DMMP values for HB-PCSOX-Phenol, HB-PCSOX-BSP3, linear phenol, and linear BSP3, which are quoted from two previous studies.<sup>12,33</sup> All three hyperbranched polyesters gave excellent responses to DMMP (Fig. 3). The strong DMMP response of HB-PE-COOH 2 was particularly



**Figure 3** SAW responses of (a) HB-PE-Phenol 1 (1759 Hz), (b) HB-PE-COOH 2 (5292 Hz), and (c) HB-PE-OH-OAc 3 (2527 Hz) to DMMP vapor.



**Figure 4** SAW responses of (a) HB-PE-Phenol **1** (1160 Hz), (b) HB-PE-COOH **2** (352 Hz), and (c) HB-PE-OH-OAc **3** (461 Hz) to DNT vapor.

noteworthy for two reasons. First, it was comparable with the response of BSP3, the linear SAW sensor polymer reported to show the greatest sensitivity towards DMMP.<sup>34</sup> Second, linear polymers carrying protic acid sensor groups such as  $\text{SO}_3\text{H}$  and  $\text{COOH}$  have been reported to perform poorly as SAW sensors.<sup>35</sup> HB-PE-Phenol **1** gave a good response to DNT

(Fig. 4), but HB-PE-COOH **2** and HB-PE-OH-OAc **3** gave poor DNT responses that were in the 400 Hz noise region. These results are in marked contrast to the DMMP results, where HB-PE-COOH **2** outperformed HB-PE-Phenol **1**. The questions of why hyperbranched COOH polymers perform better than linear COOH polymers, and of why HB-PE-COOH **2** gives the strongest response to DMMP while HB-PE-Phenol **1** gives the strongest response to DNT will be addressed in the following two sections.

### Hyperbranched versus linear architecture

The poor SAW responses of linear polymers carrying pendant protic acid groups have been attributed to the strength of intermolecular hydrogen bonding between COOH groups.<sup>35</sup> The strength of self association (i.e., enthalpy of intermolecular hydrogen bonding) has been shown to follow the order  $\text{COOH} \gg \text{PhOH} > \text{CF}_3\text{CH}_2\text{OH}$ .<sup>36</sup> For a sensing event to take place in a self-associated system, intermolecular hydrogen bonds must first be broken in order to form new hydrogen bonds with an incoming hydrogen bond-basic vapor. In a non-self-associated system, no intermolecular hydrogen bonds need to be broken for a sensing event to take place. Thus a sensing event is more thermodynamically favorable in a non-self-associated system of “free” sensor groups than in a self-associated system. Given the  $\text{COOH} \gg \text{PhOH} > \text{CF}_3\text{CH}_2\text{OH}$  order of enthalpies listed above, obviously the issue of self-association is less critical for acidic SAW polymers carrying phenol or fluorinated aliphatic alcohol sensor groups. However, in the case of HB-PE-COOH **2**, a hyperbranched architecture appears to be beneficial because in contrast to a linear polymer, its hyperbranched geometry reduces the proportion of COOH groups involved in intermolecular hydrogen-bonding. This hypothesis is supported by FTIR studies of the hydrogen-bond structure of HB-PE-COOH **2**,<sup>37</sup> where curve fitting results show both weakly and strongly hydrogen-bonded carboxylic acid groups. HB-PE-Phenol **1** was also characterized by FTIR, and shown to form a stable network of hydrogen bonds through interactions between pairs of hydroxyl groups, or between hydroxyl and ester groups, of the same or neighboring molecules.<sup>38</sup> After annealing, a better ordered and more thermally stable framework with higher density (increased refractive index) is formed using some of the acidic surface hydroxyl groups. Unfortunately in FTIR studies of both of these HB-PE systems, hydrogen-bonding for terminal and internal OH (or COOH) groups could not be distinguished.

### Hydrogen-bond acidity and polarizability

When rationalizing trends in SAW response data, it is worth noting percent weight OH content (since

**TABLE II**  
**Advancing Water Contact Angles and Isoelectric Points of HB-PE-Phenol 1, HB-PE-COOH 2, and HB-PE-OH-OAc 3 Layers**

Polymer	Advancing water contact angle (°)		Isoelectric point IEP (pH units)	
	Untreated	Annealed <sup>a</sup>	Untreated	Annealed <sup>b</sup>
HB-PE-Phenol 1				
Layer 1 <sup>c</sup>	54.0	75.1	4.19	4.52
Layer 2 <sup>d</sup>	62.4	88.4	3.7	4.0
HB-PE-COOH 2				
Layer 1 <sup>c</sup>	57.7	69.9	3.3	3.5
Layer 2 <sup>d</sup>	61.1	87.6	2.5	2.9
HB-PE-OH/OAc 3				
Layer 1 <sup>c</sup>	74.2	–	4.2	4.6
Layer 2 <sup>d</sup>	77.2	78.8	4.1	4.0

<sup>a</sup> 2 h/240°C/air.

<sup>b</sup> 1 h/240°C/air for 1 and 2, 1 h/180°C/air for 3.

<sup>c</sup> Spin-coated from 1 wt % THF solution/20 s/3000 rpm:  $n_{630 \text{ nm}} = 1.630$ , thickness 30–40 nm.<sup>25,39,40</sup>

<sup>d</sup> Spin-coated from 2 wt % THF solution/30 s/2000 rpm:  $n_{630 \text{ nm}} = 1.599$ , thickness ~ 80 nm.<sup>37</sup>

density of sensing groups relates to sensitivity), glass transition temperature (since  $T_g$  relates to crystallinity and vapor diffusion into the SAW coating), contact angle (since this relates to the ability of the SAW coating to be wetted by analyte vapor), and isoelectric point (IEP) (since this relates to the acidity of the surface). Table II shows how contact angles and IEPs vary for HB-PE-Phenol 1, HB-PE-COOH 2, and HB-PE-Phenol OAc 3 layers prepared using different spin coating conditions.<sup>25,37,39,40</sup>

Even more importantly, the compatibility between the analyte vapor and the SAW polymer must be considered. This can be treated quantitatively using linear solvation energy relationship theory,<sup>7</sup> where a solubility property such as  $K_p$  (the vapor–polymer partition coefficient) is expressed as a function of solute solubility parameters (SSPs), where  $R_2$  (excess molar refraction) corresponds to polarizability of  $n$  and  $\pi$  electrons,  $\pi_2^H$  corresponds to dipolarity,  $\alpha_2^H$  corresponds to hydrogen-bond acidity,  $\beta_2^H$  corresponds to hydrogen bond basicity, and  $L$  corresponds to a reference (*n*-hexadecane) gas–liquid partition coefficient [eq. (1)].

$$\text{Log } K_p = c + rR_2 + s\pi_2^H + a\alpha_2^H + b\beta_2^H + l \text{ Log } L \quad (1)$$

SSPs can be measured experimentally using techniques such as gas liquid chromatography,<sup>41–43</sup> and give an indication of the thermodynamic driving force necessary for a vapor to dissolve in a SAW polymer and for a strong SAW response to occur. Table III shows literature SSPs for phenol and benzoic acid<sup>44</sup> (serving as models for HB-PE-Phenol 1 and HB-PE-COOH 2 respectively), and for DMMP itself

and nitrobenzene<sup>7</sup> (serving as a model for DNT).  $pK_a$ 's (water, 25°C) for phenol and benzoic acid<sup>45</sup> are also included in Table III so that protic acidity and hydrogen bond acidity may be compared.

It can be seen from Table III that phenol and benzoic acid have comparable hydrogen bond acidities and dipolarities, but that benzoic acid has lower polarizability than phenol. It can also be seen that DMMP has high hydrogen bond basicity in combination with low polarizability, whereas DNT has lower hydrogen bond basicity in combination with high polarizability. Hence when faced with two SAW polymers of comparable hydrogen bond acidity, DMMP has the most favorable thermodynamic interaction with the one of lowest polarizability, i.e., HB-PE-COOH 2, while DNT has the most favorable thermodynamic interaction with the one of highest polarizability, i.e., HB-PE-Phenol 1. The poor DNT responses of the four nonaromatic phenolic polymers in Table I (HB-PCSOX-Phenol, HB-PCSOX-BSP3, linear phenol and linear BSP3) reflect the lower polarizability

**TABLE III**  
**Literature Solute Solubility Parameters<sup>7,44</sup> and  $pK_a$  Values (water, 25°C)<sup>45</sup> for Phenol, Benzoic Acid, DMMP, and Nitrobenzene**

	Phenol	Benzoic acid	DMMP	Nitrobenzene
$pK_a$	9.99	4.20	–	–
$R_2$ polarizability	0.805	0.730	0.220	0.871
$\pi_2^H$ dipolarity	0.89	0.90	0.83	1.10
$\alpha_2^H$ hydrogen bond acidity	0.60	0.59	0	0
$\beta_2^H$ hydrogen bond basicity	0.30	0.40	0.81	0.27

of these carbosiloxane- and siloxane-based polymers relative to the wholly aromatic hyperbranched polyesters.

## CONCLUSIONS

Hyperbranched polyesters carrying internal and exo-presented hydrogen-bond acidic functional groups give strong SAW sensor responses to vapors such as the nerve agent simulant DMMP and the explosives simulant DNT. A COOH-functionalized hyperbranched polyester gave an exceptionally strong response to DMMP. This was attributed to a lower degree of intermolecular hydrogen bonding (self-association) in the hyperbranched architecture than in a linear architecture, making a sensing event more thermodynamically favorable in the HB SAW polymer system (since fewer intermolecular hydrogen bonds needed to be broken before new hydrogen bonds could form between SAW polymer and analyte vapor). While the COOH-functionalized HB polymer gave the strongest response to DMMP, the phenol-functionalized HB polymer gave the strongest response to DNT. This was attributed to variations in polarizability SSPs for the two HB polymers and the two vapors, where phenol-functionalized HB polymer and DNT had greater polarizability than COOH-functionalized HB polymer and DMMP.

The loan of the 500 MHz SAW units, test equipment, and vapor generator used in this study from BAE Systems (Austin, Texas) is gratefully acknowledged.

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