



Cite this: *New J. Chem.*, 2023,
47, 15021

Pillar[5]arenes-based high- T_g thermosets for the capture of volatile organic compounds†

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Volatile organic compounds (VOCs) continue to be one of the biggest problems for air quality and it is becoming increasingly urgent to find new systems capable of limiting their concentration in the atmosphere. Thanks to their capacity to recognize halogenated derivatives, Pillar[5]arenes were used to form high performance thermosets able to absorb chlorine and bromide-based VOCs. Initially, poly(2-vinyl-4,4-dimethylazlactone) featuring different chain length were synthesized and modified by ring opening reaction by mono-amino substituted P[5]A. The high- T_g polymers obtained were then crosslinked by isophore diamine to form high performance materials featuring T_g up to 150 °C and storage modulus G' up to 85 MPa. The ability of P[5]A cavities to capture VOCs was investigated by vapor sorption tests in presence of dichloromethane, 1,2-dibromoethane, dibromochloromethane and 1,1,2,2-tetrachloroethane. The P[5]A-based thermosets shown to be efficient to absorb these carcinogenic VOCs and, this efficiency depends on the affinity of cavities for the studied VOCs, proving that these new generation of thermosets seems to be selective towards certain VOCs.

Received 25th May 2023,
Accepted 28th June 2023

DOI: 10.1039/d3nj02422a

rsc.li/njc

Introduction

Volatile organic compounds (VOCs) are chemicals featuring low vapor pressures and are potentially responsible for negative effect on human health and environment.¹ Outdoor VOCs pollution contributes to the “bad” tropospheric ozone and photochemical smog.^{2,3} VOCs have also other environmental impacts such as soil and groundwater contamination.⁴ Indoor VOCs are highly toxic and can cause cancer and building-related illnesses (e.g. asthma, inhalation fever...).^{5,6} These gases are emitted into the atmosphere from anthropogenic (e.g. domestic and industrial wastes or paints, fossil fuel combustion...) but also from biogenic (e.g. emission from vegetations, volcanos, sea salt...) sources.^{7,8} To summarize, VOCs constitute one of the major concerns for air-quality but they continue to be inevitable. To address this problem, technologies such as destruction,⁹ radiolysis,¹⁰ biofiltration¹¹ or capture¹² have been investigated in recent years. Among these technologies, the capture remains the simplest, the cheapest and the most sustainable method to decrease the VOCs concentration in air.¹³

Nowadays, a plethora of absorbents have been described in the literature, such as metal-organic frameworks,¹⁴ deep eutectic solvents,¹⁵ activated carbons,¹⁶ various functionalized polymers^{17,18} or fibers.^{12,19} All these technologies are clearly focused on their VOC absorption capacity but only a few of them have actually been integrated into an inexpensive material that can be easily handled, transported, stored and recycled. In this vein, proposing absorbent materials based on common plastics having high glass-transition temperature (T_g) and/or high storage modulus would certainly constitute an interesting strategy. Currently, only hypercrosslinked polymers (HCP) feature these characteristics.²⁰ Mostly synthesized by Friedel–Crafts alkylation reaction, the extensive crosslinks give them a permanent porosity with high specific area (up to 1000 m² g^{−1}) allowing to absorb efficiently different groups of VOCs such as aromatic hydrocarbons or ketones.^{21,22} Although this strategy is remarkably successful, the pore size distribution in HCP cannot easily be controlled, resulting in absorbents which usually lack of selectivity.²³ Thus, in this work, we examine whether a controlled microporosity can be controlled by installing rigid cavities of a given size within thermosetting polymers? Our attention turned to macrocycles-based systems which have been described in the literature as absorbents of pollutants.^{24–26} Among those, modified pillar[5]arenes, supramolecular receptors composed of hydroquinone units,²⁷ have demonstrated high efficiency to recognize pollutants or herbicides.^{28–31} Pillar[5]arene (P[5]A) are very rigid and the aromatic constituents in their structure leads to the formation of strong

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† Electronic supplementary information (ESI) available. See DOI: <https://doi.org/10.1039/d3nj02422a>



complexes by π -H or π - π stacking with electron-poor guests such as paraquat, pyridinium and halogenated derivatives.^{32,33} Therefore, we propose to create a crosslinked material featuring high T_g and containing grafted P[5]A. In the past, copolymerization of P[5]A-based monomers was reported,^{34,35} here we propose an alternative approach based on the post-functionalization of poly(2-vinyl-4,4-dimethylazlactone) (PVDMA). PVDMA was chosen due to its capacity to be easily modified by ring-opening reaction using alcohol or amine functions.^{36,37} This strategy allowed us to obtain polymers with 50% of P[5]A as pendant groups *via* a simple reaction devoid of by-products. After neutralization and crosslinking with isophorone diamine (IPD), high- T_g thermosets were obtained which capture halogenated derivatives, leading to a mass increase as high as 33 wt%. Thus, we believe that this strategy we have devised constitutes an interesting proof of principle that materials with controlled microporosity can be employed as efficient VOC absorbents.

Results and discussion

Syntheses and formation of crosslinked materials

First of all, the 2-vinyl-4,4-dimethylazlactone (VDMA) monomer and the mono-amino Pillar[5]arene (P[5]A-NH₂) were synthesized in a two-step and a four-step reaction respectively according to the procedures already described in the literature^{38–40} and detailed in ESI† with the corresponding NMR spectra (Fig. S1–S6). Then, VDMA was polymerized by Reversible Addition-Fragmentation Chain Transfer (RAFT) polymerization to obtain well-defined polymers. Two different chain lengths were targeted in order to assess the impact of the chain-length on the final T_g and also on the VOCs' capture tests. The two PVDMA samples, characterized by nuclear magnetic resonance (NMR) spectroscopy, size exclusion chromatography (SEC, Fig. S13 and S14, ESI†) and differential scanning calorimetry (DSC, Fig. S15, ESI†), have respectively a number average molecular weight of 12 400 and 36 200 g mol^{−1} with dispersities as low as 1.3 and T_g of 124 °C and 182 °C (Table 1). Polymers with lower T_g were not retained and polymers with $T_g > 200$ °C proved too brittle to be in accordance with the objective of this study and the formation of commodity thermosets.

Then different percentages of P[5]A-NH₂ were grafted onto PVDMA as pendant group. As aforementioned, the azlactone rings are known to be easily opened by amine functions, forming stable amide functions (Fig. 1). These amides are more

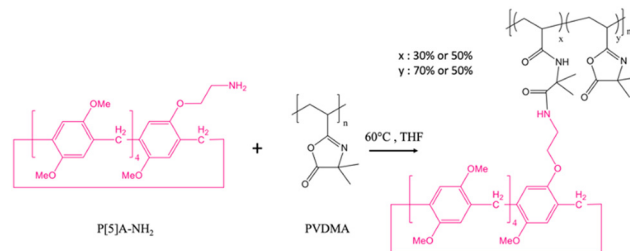


Fig. 1 Functionalization reaction of 30 or 50% P[5]A-NH₂ on PVDMA chains.

difficult to hydrolyze than ester functions we could have obtained if we had decided to use mono-hydroxyl P[5]A.

A DOSY NMR analysis was used to identify the extent of grafting. Indeed, for a PVDMA post-functionalized by 50% of P[5]A-NH₂, an identical diffusion coefficient was observed for both polymer chains and P[5]A-NH₂ confirming the presence of a single compound (Fig. S18, ESI†). On the other hand, for a PVDMA containing 60% of P[5]A-NH₂, two different diffusion coefficients were observed for the P[5]A-NH₂ peaks, proving that a low percentage was not grafted onto the polymer chain (Fig. S19, ESI†). The failed attempt to reach P[5]A-grafting higher than one out of two VDMA units is probably due to the steric hindrance resulting from bulky P[5]A structure.⁴¹ Beyond DOSY NMR, a SEC chromatography was performed to prove the modification of the polymer and an increase of its number molecular weight average. As an example, Fig. 2 shows a polymer post-functionalized with 50% of P[5]A-NH₂ and the PVDMA from which it was derived.

Once the grafting of P[5]A-NH₂ was completed, it was necessary to neutralize the rest of the azlactone rings unreacted in the chain. For this neutralization, butylamine was selected among other candidates such as dodecylamine. Indeed, it is a good compromise allowing to reduce the possibility to create complex between the alkyl chain and the P[5]A cavity, compared to dodecylamine.⁴² After reaction with butylamine, only 10 mol% of azlactone rings

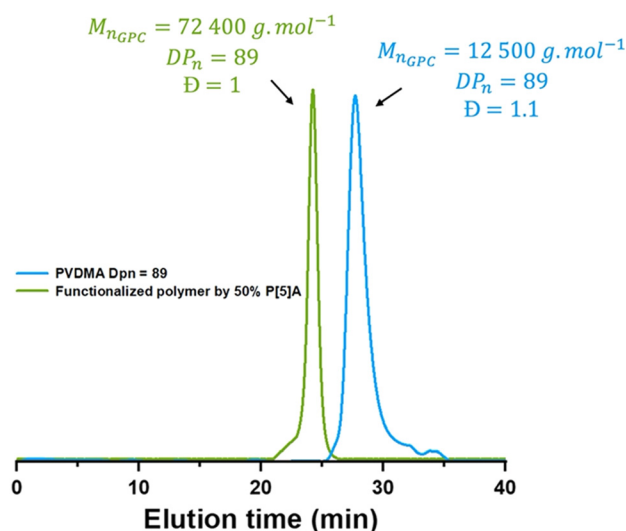


Fig. 2 SEC results obtained for PVDMA DP_n = 89 (blue) and after functionalization by 50% P[5]A-NH₂ (green).

Table 1 Glass transition temperature (T_g) of crosslinked materials

| Polymer or material | DP _n (SEC) | % P[5]A | % Butylamine | % IPD | T_g (°C) |
|---------------------|-----------------------|---------|--------------|-------|------------|
| PVDMA 1 | 89 | 0 | 0 | 0 | 124 |
| PVDMA 2 | 261 | 0 | 0 | 0 | 182 |
| 1-0 | 89 | 0 | 90 | 10 | 132 |
| 1-30 | 89 | 30 | 60 | 10 | 144 |
| 1-50 | 89 | 50 | 40 | 10 | 135 |
| 2-0 | 261 | 0 | 90 | 10 | 134 |
| 2-30 | 261 | 30 | 60 | 10 | 152 |
| 2-50 | 261 | 50 | 40 | 10 | 147 |



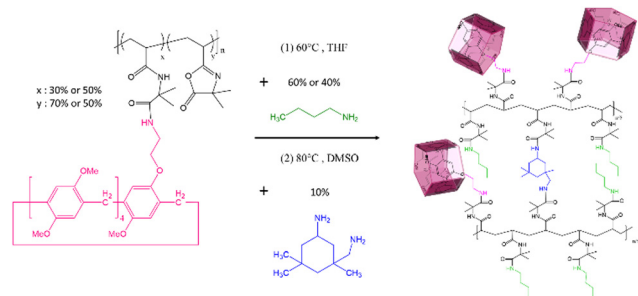


Fig. 3 Neutralization and formation of thermosets with addition of IPD (10 mol%).

were left unreacted, which could be used for crosslinking (Fig. 3). Therefore, the crosslinking step consisted in the addition of 10 mol% of isophorone diamine (IPD). IPD was chosen as crosslinker for its low toxicity regarding human health and the environment.⁴³ At the end, four thermosets were finally obtained from the two initial PVDMA. Indeed, two different percentages of P[5]A-NH₂ (30% and 50%) were grafted onto the two different chain lengths PVDMA and so, 60% or 40% of butylamine were added to neutralize the other azlactone cycles finishing by the crosslinking process.

All these thermosets were characterized by DSC like their intermediates (Table 1 and Fig. S15, S16, ESI[†]). Regardless of the chain length of the precursor polymer or the percentage of P[5]A, relatively close *T_g* were determined (between 135 and 152 °C). By adding pendant groups like P[5]A and butylamine, an expected decrease of *T_g* was observed (Fig. S15, ESI[†]) and a light increase was logically obtained after the crosslinking.

VOC capture ability

The VOC capture capacity of these high-*T_g* thermosets was investigated with four different VOCs, namely (i) the dichloromethane (A), (ii) the 1,2-dibromoethane (B), (iii) the dibromochloromethane (C) and (iv) the 1,1,2,2-tetrachloroethane (D). Indeed, these chlorine and bromine VOCs are known to be toxic and carcinogen for humans or animals and are, therefore, among the most dangerous VOCs for the health and environment.^{44,45}

A simple calculation allowed us to determine the theoretical absorption percentage that the P[5]A can allow (eqn (1)). Note that we use the word “absorption” because VOCs are capture in the bulk of the material, but the word “adsorption” is equally used in literature to refer to the same phenomenon. Eqn (1) works if we take into consideration that, according to the literature, only one VOC molecule can be recognized by one P[5]A cavity (1 : 1 host/guest complex).

$$\%_{\text{theo adsorp. by P[5]A}} = \frac{nb_{\text{P[5]A}} \times M_{\text{VOC}}}{M_{\text{chain}}} \times 100 \quad (1)$$

In the eqn (1), $nb_{\text{P[5]A}}$ represents the number of P[5]A per chain, M_{VOC} and M_{chain} represent the molecular weight of the VOC captured and of the polymer chain respectively.

As the chain length is not relevant to this calculation, we obtained the same results for materials 1–30 and 2–30 or for 1–50 and 2–50. Indeed, for materials containing 30% of P[5]A,

Table 2 Maximum theoretical absorption percentage of VOCs determined by eqn (2)

| Material | VOC A | VOC B | VOC C | VOC D |
|---------------|-----------------|------------------|------------------|-----------------|
| 1–0 and 2–0 | 1% ^a | 15% ^a | 19% ^a | 2% ^a |
| 1–30 and 2–30 | 7% | 28% | 33.5% | 14% |
| 1–50 and 2–50 | 8.5% | 31.5% | 37% | 17% |

^a Determined experimentally by vapor sorption after 14 days.

theoretical absorption percentages of 6%, 13%, 14.5% and 12% were determined for VOC A, B, C and D respectively while 7.5%, 16.5%, 18% and 15% were calculated in case of 1–50 and 2–50. However, the theoretical absorption percentage of the materials is not fully taken into account in eqn (1) as PVDMA alone (*i.e.* devoid of P[5]A) can absorb VOC vapors. This absorption was determined experimentally by testing materials 1–0 and 2–0 under VOCs (A, B, C and D) atmosphere. After 1 days of vapor sorption, plateaus were observed for each VOCs (Fig. S24–S29 and Table S1, ESI[†]) and no significant difference was observed with the increase of the chain length. Finally, the maximum theoretical absorption percentage of the materials could be predicted (eqn (2)) by adding the amount absorbed in the polymer to the amount calculated from eqn (1) (Table 2):

$$\%_{\text{theo adsorp. max.}} = \%_{\text{theo adsorp. by P[5]A}} + \%_{\text{exp. adsorp. without P[5]A}} \quad (2)$$

Experimentally, vapor sorption was performed for each VOC. For this, each material was previously dried under vacuum for one night to avoid possible presence of other solvents in the material before the analysis. The dried thermosets were then suspended in a vial containing 1 mL of VOC. The materials were then weighed every day to determine the weight change and extrapolate the experimental absorption percentage of VOC.

First of all, the impact of the chain length was investigated. Vapor sorption of A and D was performed by using 1–30 and 2–30. At most, 2% deviation was observed between both materials (Fig. S24 and S25, ESI[†]). Whether in terms of thermal or sorption properties, the chain length has no impact.

Then, the impact of the P[5]A cavities was verified (Fig. 4): for all VOCs, the amount of absorbed VOC increases with the amount of P[5]A contained in the material. For the case of VOC A by the material 2–50 (red points on Fig. 4(a)), it takes 4 days to saturate the material with VOC: at this point, the mass increase is of 8.5%, which compares well with the 7.5% predicted by eqn (2) (shown by the dotted line in Fig. 4a). The same observation can be made for the material 2–30 and for the VOCs C and D. However, in the case of VOC B, the plateau occurs after 7 days and an absorption percentage of 53% was observed instead of the predicted 31.5%. As P[5]A and B (1,2-dibromoethane) forms a 1 : 1 complex (Fig. S22 and S23, ESI[†]), this result can be justified by the formation of sandwiches within the system.^{46,47} Indeed, B can form a complex with P[5]A featuring a high K_a up to 2 900 M^{−1} while A forms a much weaker complex ($K_a = 120 \text{ M}^{-1}$), as determined in literature.⁴⁸ This strong affinity between the cavities and B can lead to the



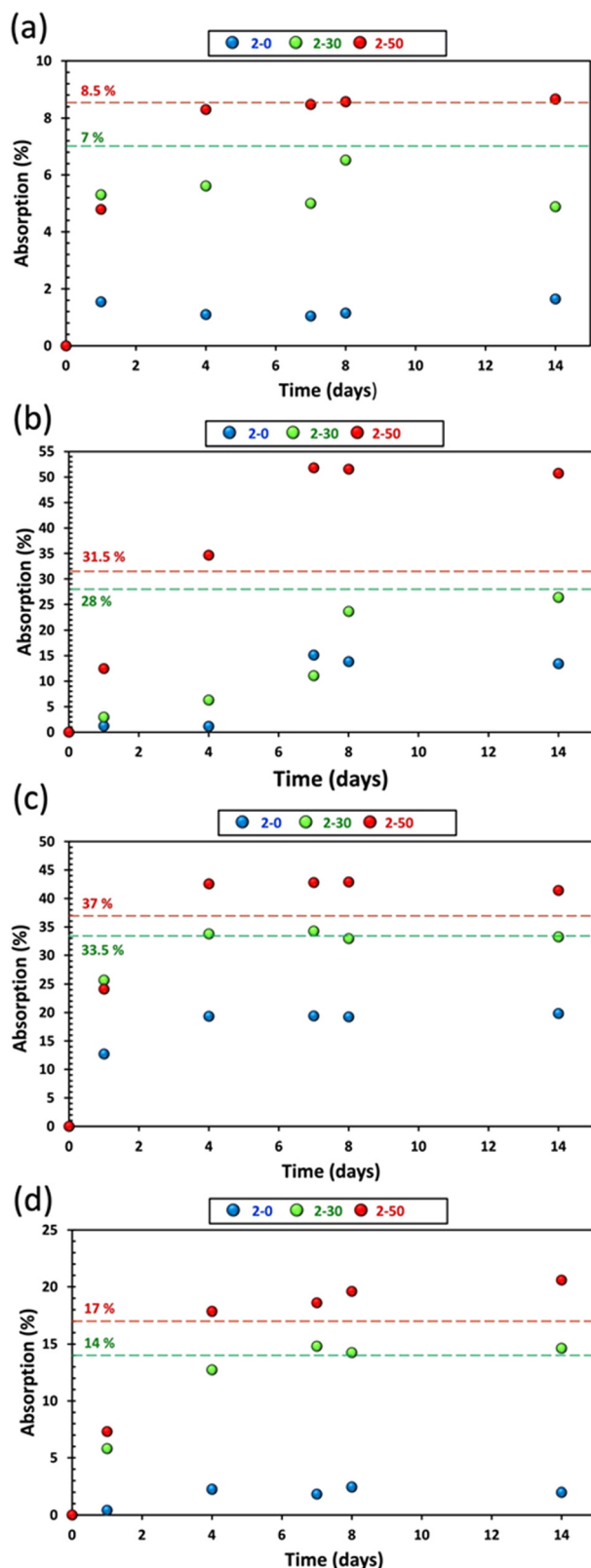


Fig. 4 Absorption percentages of VOC A (a), B (b), C (c) and D (d) by 2-0, 2-30 and 2-50. The absorption percentages are the average of triplicate tests. 20 mg of materials and 1 mL of VOC were used for tests.

formation of a super structures where a molecule of **B** is trapped between two filled cavities, like a sandwich. This self-assembly can occur when the cavities are sufficiently close to each other like in the 2-50. But, in the case of 2-30, the cavities are too far away, the sandwich cannot be formed and therefore, the measured absorption percentage is in good agreement with the one predicted by eqn (2).

All these results confirm that these thermosets have the capacity to capture VOCs, in amounts which depends both on the percentage of grafted P[5]A and on the affinity between the P[5]A and the VOC.

More interestingly, the selectivity factors observed with these thermosets are higher than other systems described in the literature. Indeed, factors up to 6 between VOC **B** and VOC **A** (Fig. S34, ESI†) were observed while pillared clay-based systems proposed by Pires *et al.* showed factors lower than 1.5 between two halogenated hydrocarbons.⁴⁹ Another essential study, to correlate the thermosets to future applications, is to determine if the captured VOCs remain within the material or if they are released over time.

To realize this study, 10 mg of VOCs were first captured by 2-30 and 2-50. After 7 days in open air, the mass of the remaining mass of the materials were determined (Fig. 5). As a control sample, 10 mg of VOCs were disposed in a tared vial and weighed after 7 days.

Without materials, all VOCs were evaporated. All the dichloromethane (**A**) was released from the different materials. This can be explained by the low association constants found in the literature between **A** and P[5]A compared to bromine derivatives⁵⁰ and by the low boiling point of **A**. By contrast, the three other VOCs remained trapped in the materials, since more than 80% of mass were retained after 7 days.

It should be noted that in the case of VOC **B**, the 2-50 material retained less VOC than 2-30. This may be assigned again to the presence of sandwich-type structures. Indeed, the VOCs trapped between two different cavities are not as strongly retained as those contained in the cavities and are therefore released faster.

Moreover, the reusability of the materials for capturing VOCs was investigated using VOC **A** as the model as its removing under vacuum was more efficient than the other VOC.

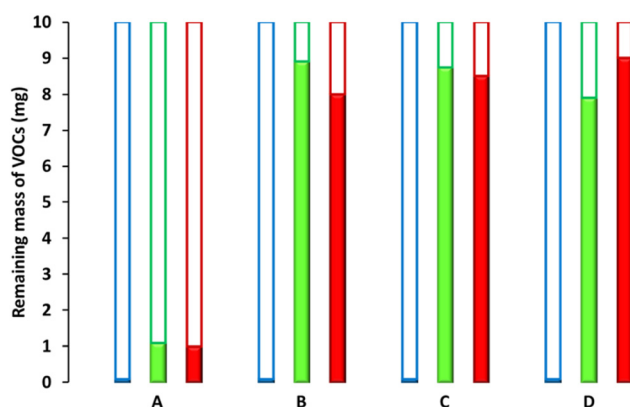


Fig. 5 Remaining mass of VOCs **A**, **B**, **C** and **D** in materials 2-30 (green) and 2-50 (red) and without material (blue) after 7 days in open air.



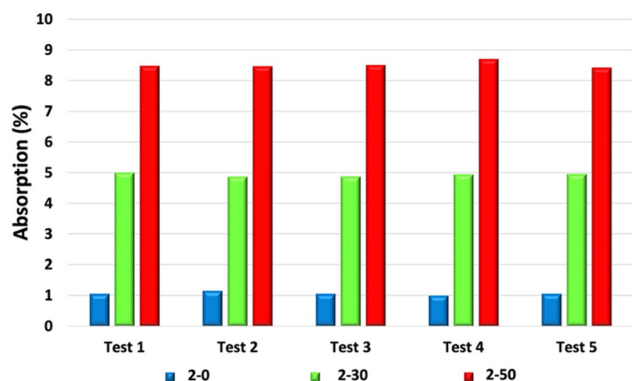


Fig. 6 Reusability of materials 2-0, 2-30 and 2-50 for capturing VOC A.

For this, the materials were placed in a vacuum oven overnight and they were placed for 7 days under A atmosphere. It can be seen that, after five absorption cycles, the capture ability of the three materials 2-0, 2-30 and 2-50 for A vapor showed no obvious change (Fig. 6), demonstrating their good reusability for capturing VOCs.

In addition, an analysis allowing to observe the impact of the VOC on the thermal properties of the material was carried out. For this purpose, the material 2-50 placed in a B atmosphere was chosen. Indeed, being the material capturing the most quantity of VOCs, it is then likely to see its thermal properties the most impacted. By comparing the DSC of the material before ($T_g = 147^\circ\text{C}$) and after ($T_g = 144^\circ\text{C}$) absorption, no change of the thermal properties was observed since the T_g remains identical (Fig. S17, ESI†).

Thermo-mechanical properties

Finally, the thermo-mechanical properties of these materials were determined by shear DMTA (Fig. 7 and Fig. S33, ESI†). The materials feature interesting thermo-mechanical properties with T_α values measured at the peak of $\tan \delta$ higher than 160°C , in agreement with T_g values obtained by DSC and a shear modulus (G') at room temperature up to 30 MPa for both materials. These thermo-mechanical properties are similar to usual thermosets.⁵¹

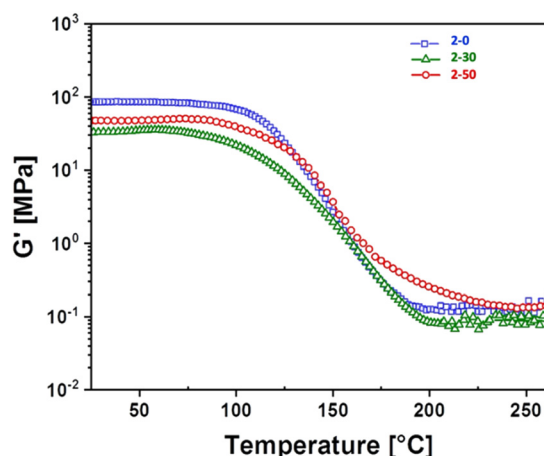


Fig. 7 Storage modulus G' versus temperature of materials 2-0 (□), 2-30 (Δ) and 2-50 (○) obtained by shear DMTA.

Conclusions

A new type of Pillar[5]arene-based thermosets was synthesized *via* the polymerization of 2-vinyl-4,4-dimethylazlactone followed by a ring-opening procedure to avoid the formation of side products. These thermosets featured high mechanical and thermal properties and showed their efficiency in the capture of halogen-based VOCs thanks to the recognition ability of P[5]A. Interestingly, materials with high percentage of P[5]A were found to be more effective for certain VOCs, such as dibromoethane, which showed a strong affinity for P[5]A and, therefore, allowed the formation of strong supramolecular complexes and sandwiches to keep the VOC in the thermosets. This leads us to believe that these materials, comparing to hypercrosslinked polymers for example, can be selective towards certain VOCs. These materials conserve their desirable mechanical properties, even when filled with VOC, indicating that the VOCs do not plasticize the polymer. Moreover, even if these materials were able to retain more than 80% of the captured VOC after one week, they can be easily regenerated and reused. We hope that this study will open the door to a new family of eco-friendly thermosets. Moreover, in future works, the selectivity of these materials will be carefully considered by preparing thermosets featuring larger P[n]A cavities to obtain a wider range of materials adapted to the environmental demands. Finally, we intend to test these materials in a real tested for air purification.

Author contributions

SR: writing original draft, data curation and analysis, VT: data curation and analysis, PW: supervision and review and editing, JC and JP: formal analysis, supervision, writing review and editing.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

The authors thank Dr V. St-Onge for help with the DMA. S. Ritaine thanks Université de Lille for a fellowship. Pr. J. Clavier acknowledges funding from the Canadian Foundation for Innovation, Canada Research Chairs and the Natural Science and Engineering Research Council. P. Woisel and J. Potier thank the project ARCHI-CM, Chevreul Institute (FR 2638), Ministère de l'Enseignement Supérieur, de la Recherche de l'Innovation, Région Hauts-de-France and European Regional Development Fund (FEDER) for funding this work.

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