Breakthrough Studies of Methyl Salicylate and DMMP Mixed in Methyl Salicylate with Pressure Swing Adsorption Composed of 13X Molecular Sieves

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ABSTRACT

A test procedure for pressure swing adsorption (PSA) was established and elucidated for the air purification using methyl salicylate (MeS) and 5 per cent (v/v) dimethyl methyl phosphonate (DMMP) in MeS containing air stream as feed. The effect of feed flow rate was studied by varying the flow from 5 lpm to 20 lpm, for both the molecules at 25 ℃ and 4 kg/cm². The results revealed that the flow rate had a significant influence on the breakthrough time. A method was developed for the determination of feed, purge, and dry air composition, by the solvent extraction method using the XAD-2 and the average concentrations reported. The 13X molecular sieves were characterised for its structural and textural properties such as BET- SA, XRD, and FT-IR. The temperature programmed desorption of DMMP and MeS on 13X clearly demonstrated that it was easily regenerated at ~320 °C after prolonged field operation of PSA. The PSA results obtained with PSA composed molecular sieves appeared to give promising technology for air purification and specifically to the chemical warfare agents simulants.

Keywords: Methyl salicylate, dimethyl methyl phosphonate, pressure swing adsorption, 13X molecular sieves, XAD-2

1. INTRODUCTION

Even though the production and possession of toxin weapons are banned by the weapon convention, yet, there are documented instances of their usage. Confronted with the problem of chemical and biological warfare agents caused all over the world, further, threat of going these warfare agents into the hands of terrorists causes fear to the mankind. Moreover, emerging trends in the information technology create easy accessibility for production and use of these chemicals by individuals and non-State actors. The early detection and protection against chemical warfare agents (CWAs) exposure can be accomplished, when armed with appropriate equipments. A number of systems are available for individual and collective protection to support armed forces from a chemical threat. In general, the whetlerite carbon together with high-efficiency particulate air (HEPA) is commonly used for nuclear, biological, and chemical (NBC) filtration systems as filtration media. HEPA provides the protection against particulates and aerosols; whereas, impregnated carbon for persistent and non-persistent chemical warfare agents. In addition, filter life depends on the exposed contaminant concentration and nature of the agent. These filtration systems are sensitive to the moisture, which demand frequent replacements even without exposure to the agents and cause a logistic and economic burden. Hence, there is a need to develop regenerable filters with longer life and minimal maintenance.

Although industrial usage of pressure swing adsorption (PSA) is widespread, with the expanding PSA applications in the air purification of NBC agents, it has become essential to optimise the design and operation of PSA. Owing to the low operating costs and high performance, PSA is considered as a promising technology to reduce the logistic burden of NBC filters.

The PSA process based on the physical binding of gasses to adsorbent material and the performance depends on the type of adsorbent material, gas component, partial pressure, etc[1]. The technology has been demonstrated in various applications such as hydrogen purification, biogas upgrading, CO_2 removal, air separation, onboard oxygen generation, etc[2-9].

Molecular sieves are widely used as adsorbents in PSA for gas separation and purification processes and are particularly suitable for CO_2 capture, H_2 recovery, with majority of the studies dealing with the modelling[10-15]. Though the engineered PSA systems were well developed, the testing of these systems with CWAs and simulants is still challenging to ascertain the process at trace level. The efforts are directed to develop air filtration systems for the removal CWAs simulants, by using PSA, and also the feed stream generation and test method using Methyl salicylate (MeS) and 5 per cent (v/v) dimethyl methyl phosphonate (DMMP). These two chemicals are widely accepted as simulants for blister and nerve agent respectively.

2. MATERIALS AND METHODS
2.1 Characterisation of Molecular Sieves

The 13X molecular sieves, 1-1.5 mm spherical beads (M/S Sorbead India Pvt Ltd) used as an adsorbent were characterised for their physical properties like BET-SA, BJH pore size distribution, and X-ray diffraction. The N_2-BET surface area

was 746 m²/g and adsorption and desorption isotherm profile (Fig. 1(a)) confirmed the microporous nature of 13X. The pore diameter (Fig. 1(b)) of 8.15Å is well acceptable for the removal of the majority of CWAs and simulants[16].

The diffraction signals (Fig. 2) at 2θ = 10°, 11.6°, 15.4°, 20°, 23.3°, 26.7°, 29.3°, and 31° correspond to the typical pattern of crystalline 13X matching with FAU structure[17]. The FT-IR spectra of 13X shows the spectral bands around 3479 cm⁻¹, 1000 cm⁻¹, 672 cm⁻¹, and 464 cm⁻¹ corresponding to the vibrations of Si-O-H, internal tetrahedral unit and the bands around 1100 cm⁻¹, 758 cm⁻¹, and 565 cm⁻¹ are ascribed to pore opening and external linkage vibrations[18]. The band at 1626 cm⁻¹ is ascribed to bending frequency of surface adsorbed H_2O.

2.2 Feed Stream Generation and Quantification

The high pressure contaminated feed (HPCF) generation system (SS 316, 60 cm H, 8 cm dia) was designed and interfaced between the compressor and the PSA system and schematic set-up is depicted in Fig. 3. This vessel is partitioned into upper and lower sections by a perforated mesh, with the upper one filled with the glass beads and lower one (vol 1.5 l) with liquid (MeS or 5 per cent (v/v) DMMP in MeS). The vessel has one inlet (liquid dip tube) which is interfaced with the compressor outlet and another one that carries the contaminated air to the PSA. The vessel is thermostated at 25 °C and maintained at constant pressure. By using this vessel one can feed the saturated vapour of contaminant air with high consistency for high boiling compounds. The concentration of the PSA feed was also standardised by using the XAD-2 adsorption tube.
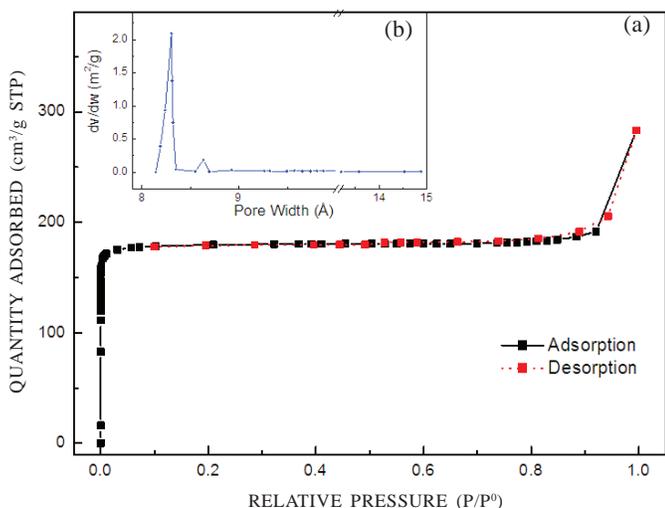
Figure 2. X-ray diffraction pattern of 13X molecular sieves.
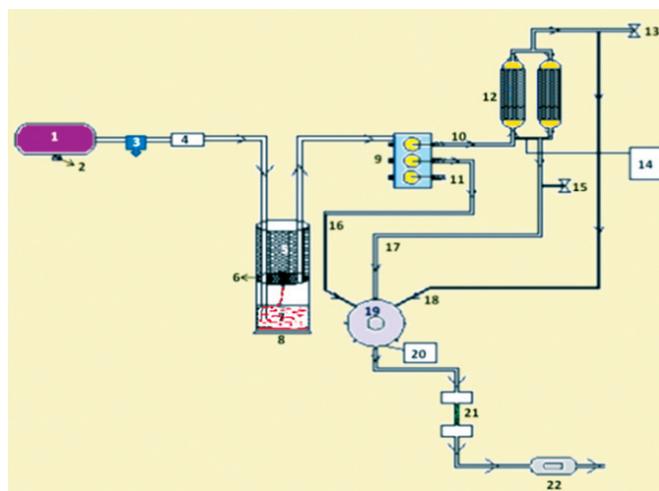
Figure 1. (a) BET adsorption and desorption isotherm (b) Pore size distribution of 13X.

2.2.1   PSA System Description

The PSA system (Fig. 3) is equipped with a displacement compressor (Frank technologies, 50 lpm) of 20 l receiver tank with pressure switch for precise pressure regulation and operates between 5-7 kg/cm². It operates with a standard time combination controller (EAPL Pvt. Ltd. India) with eight relays, and two are connected to the 5/2 way control valves. The twin tower PSA (Trident Pneumatics Pvt. Ltd, India) with standard cylindrical pipes (15 cm ht, 1.3 cm dia) can accommodate 30 g of 13X molecular sieves for each tower.
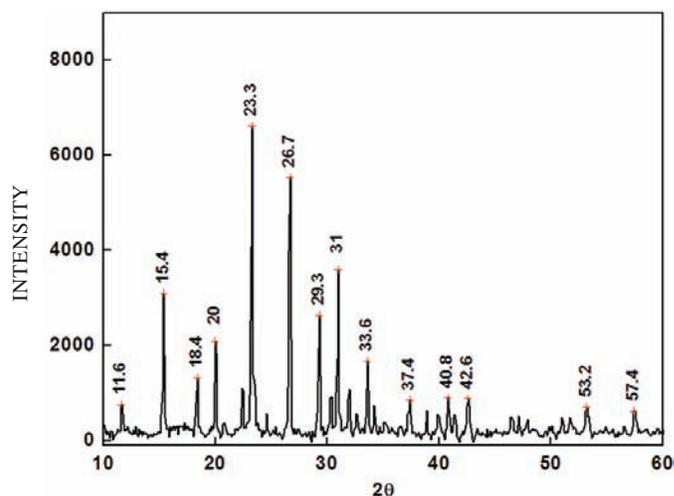
The PSA dry air flow rate is controlled by a mass flow controller (Serria instruments) and high-pressure needle valve, for the purge flow, and it is measured by a digital flow meter (TSI). The system is equipped with stream selection valve (VICI instruments) which was interfaced with compressor inlet (HPCF vessel), feed, dry air, and purge flow streams. The stream selection valve outlet has been interfaced with an XAD-2 tube with the flow controller.

2.3 Preparation of XAD-2 Tubes

The XAD-2 resin is widely used to adsorb the organic compounds from the air due to its unique macro-reticular porosity (0.41 mL PV, 90 Å PD) with a large surface area of 300 m²/g besides its stability up to 200 °C[19]. XAD-2 (Sigma-

Aldrich, 20-60 mesh) tubes were employed for analysing the PSA feed, purge, and dry air concentrations. XAD-2 resin used to prepare the sorbent tubes, which has been conditioned by triple distilled water followed by methanol, to remove all traces of preservative agents and residual monomers and further oven dried at 100 ºC prior to use. The XAD-2 tubes were made of clean glass tube of 12 cm length and 0.3 cm inner diameter. The desired amount of XAD-2 was packed with two sections of 600/300 mg. The front adsorbing section containing 600 mg will collect the compound of interest. A backup section contains 300 mg of XAD-2 to determine if the breakthrough of air born contaminants occurred on the front section, and both the sections were separated by quartz wool. In each set of experiment, a constant flow of 800 cc was maintained through an XAD-2 tube for 10 min for sampling. After sampling, the tubes were capped with the plastic caps immediately.

2.3.1 Analysis of XAD-2 Samples

The XAD-2 in each tube was transferred to a 5 ml screw-cap glass sample vial. Each tube was analysed separately using 2.0 ml of ethyl acetate. The samples were stirred vigorously using a vortex mixer (CM 101 Cyclomixer, Remi Equipments Pvt. Ltd., India) for 10 min to facilitate the desorption. The sample was transferred to the autosampler vials for GC analysis. Extracted samples were analysed by Bruker GC with PFPD/FID using DB-5 column (30 m x 0.25 mm). The oven temperature was programmed from 130 ºC to 220 ºC at the rate of 15 ºC/min. The multi-point calibration method was used for quantification of MeS and DMMP. MeS and DMMP in ethyl acetate were used as standard solvents. The peak area plotted against concentration, and the calibration curves for both the FID and PFPD detectors showed linear (Fig. 4, $R^2 = 0.998$) response against MeS and DMMP.
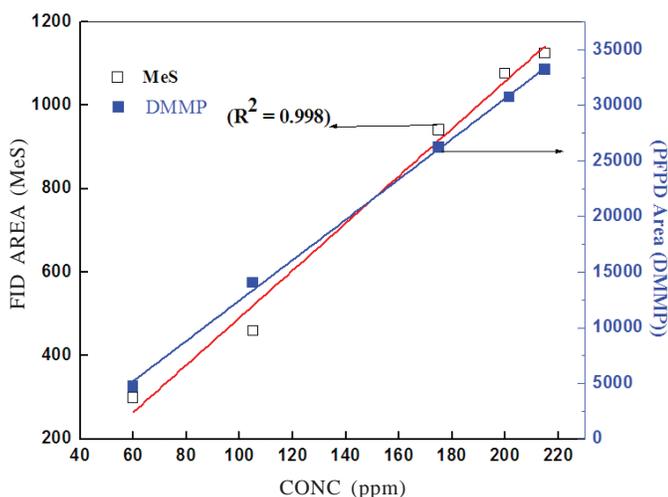

Figure 4. Calibration curves of MeS and DMMP.

3. RESULTS AND DISCUSSION
3.1 PSA Concentration Profile with Flow Rate

The MeS (1 L, Sigma-Aldrich ≥99 per cent) (Fig. 3) was filled-in HPCF vessel and interfaced with the compressor air inlet. The compressed air through a pressure regulator was gradually raised to 4 kg/cm^2 through a non-returnable valve (NRV). The outlet of the high-pressure vessel was connected to the PSA by a pressure distribution panel. The oven-dried molecular sieve was charged into the PSA tower by agitating manually and mounted on the PSA blocks. The purge air valve was closed, and the control measurements were performed for quality checks, which include the leak test.

The PSA studies were performed at various flow rates of 5 lpm, 10 lpm, 15 lpm, and 20 lpm at constant pressure of 4 kg/cm^2. For each set of experiment, purge flow was set at the 15 per cent of the total flow, which was maintained at 0.8 lpm, 1.5 lpm, 2.25 lpm, 3 lpm and the corresponding dry air flow was 4.2 lpm, 8.5 lpm, 12.75 lpm, 17 lpm, respectively. Initially, the compressor outlet flow was maintained constantly through the HPCF vessel for half an hour at 5 lpm to attain the equilibrium concentration. The cycle time was set at 10 min, i.e. each tower runs for 10 min and for each tower feed, purge, and dry air concentrations collected on XAD-2 tubes at regular time intervals within 10 min for each tower. The fixed flow of 800 ccpm was passed through the XAD-2 tube for 10 min and the five samples for analysis were taken at each concentration, and the average value was reported. The tubes were capped immediately after sampling and further analysed by solvent desorption. The extracted sample, were quantified by using calibration curve (Fig. 4) obtained by the GC. The backup section of XAD-2 (300 mg) was also subjected to analysis, and no breakthrough was observed in the sample tubes. The experiments were repeated with the 10 lpm, 15 lpm, and 20 lpm. Prior to the experiment, the compressor was drained to remove the moisture and residual contaminant, and all the test lines were flushed with fresh air to avoid any residue transfer during the experiments. Similarly, the experiments were carried out using 5 per cent (v/v) of DMMP in MeS using the same HPCF vessel filled with 1 l of 5 % (v/v) of DMMP in MeS instead of only MeS.

The PSA test results obtained at 40 min at various flow rates are depicted in Fig. 5. The variations of feed, purge and dry air concentration of PSA system as a function of flow for both the set of experiments were described. TheMeS feed concentration (Figs. 5(a) and (b)) varied from 120 ppm to 170 ppm with the increase of flow rate from 5 lpm to 20 lpm. It is interesting to observe that in both the set of experiments, MeS average feed concentrations increased marginally indicating that the influence of flow rate on feed concentrations was minimum. Upon addition of DMMP to MeS, change in the overall feed concentration was found to be minimal. Whereas, the DMMP concentrations in the feed (Fig. 5(b)) was 55 ppm to 80 ppm with a variation of flow rate from 5 lpm to 20 lpm, respectively.

The purge air concentrations in the MeS experiments, (Fig. 5(a)) varied from 150 to 500 ppm whereas in the 5 per cent (v/v) of DMMP in MeS experiments, (Fig. 5(b)) the average purge concentrations increased from 180 to 430 ppm. It is obvious that in both the cases, the purge concentrations are much higher than that of inlet concentrations. On the other hand, in the mixture, DMMP average feed concentrations (Fig. 5(b)) increased from 55 ppm to 80 ppm and average purge concentrations from 30 ppm to 80 ppm. The dry air concentrations are not detectable (found to be zero) for 40 min in all the set of experiments and neither MeS nor DMMP was identified by the FID/PFPD in the solvent XAD-2 extraction method.
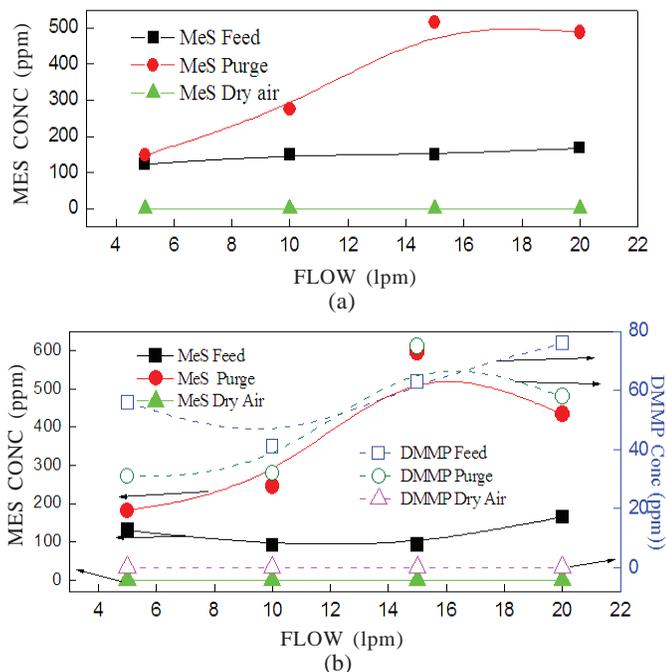
Figure 5. PSA feed, purge and dry air concentrations for 40 min (a) MeS (b) 5 per cent (v/v) DMMP in MeS.

3.2 Effect of Flow Rate on the Breakthrough Time for MeS and 5 per cent (v/v) DMMP in MeS

The breakthrough studies were performed for the flow rates of 5 lpm, 10 lpm, 15 lpm, and 20 lpm at 4 kg/cm² for PSA system. The PSA dry air concentrations were measured at regular time intervals by sorbent tube method. The concentration profiles at various flow rates as function of time is depicted in Fig. 6(a) and the MeS average inlet and purge concentrations were 150 ppm and 360 ppm, respectively.

The results revealed that with increasing the inlet flow rate the breakthrough occurred much faster and the break through time (Bt) values were 160 min at 5 lpm and the Bt value reduced to 60 min at 20 lpm (Fig. 6(b)). The early break through at higher flow rates is ascribed to (a) decrease in contact time, (b) variation in the PSA inlet concentrations. However, the influence of flow rate on the PSA feed concentrations (Fig. 5(a)) is marginal whereas, the effect of flow rate is more significant with respect to Bt values.

Similarly, the experiments were repeated with the 5 per cent (v/v) DMMP in MeS. The results revealed that no breakthrough was observed for 40 min (Fig. 6(b)). After 40 min, similar breakthrough pattern was observed for MeS in the mixture (Fig. 7) with increasing the flow rate with marginal variations in the MeS concentrations. On the other hand, it was interesting to observe for DMMP tests that no breakthrough was detected by the PFPD, which could be attributed to the relative lower abundance of DMMP than MeS, and relative polar nature of DMMP than MeS, which made the preferential binding of the DMMP with the 13X pores.

3.3 FT-IR Studies on Adsorbed 13X Molecular Sample

The strongly adsorbed components to flush back in purge step, as purified air passed in the reverse direction allows
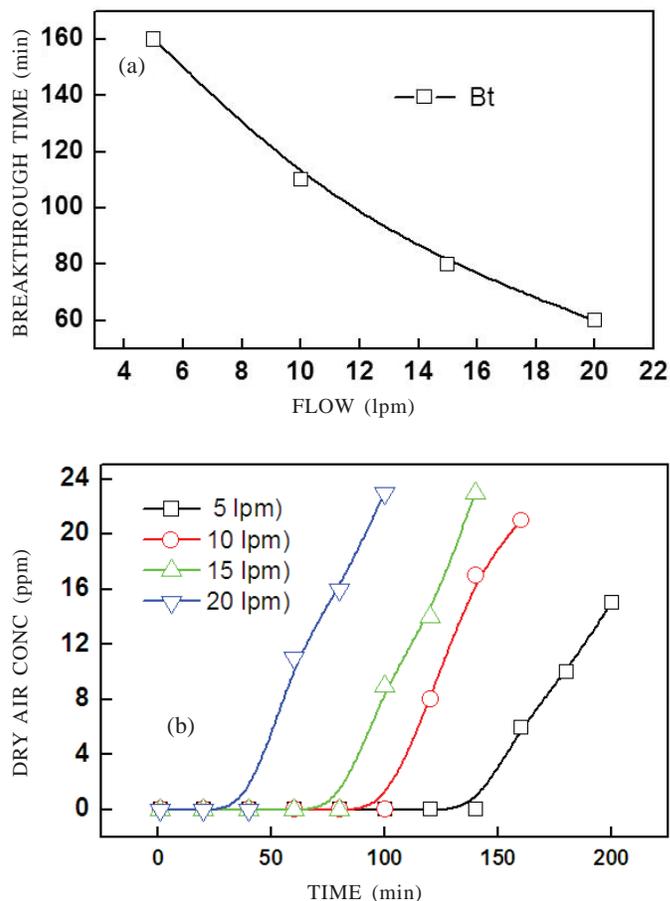

Figure 6. MeS experiment (a) Bt profile of MeS as function of flow (b) MeS concentrations in dry air.
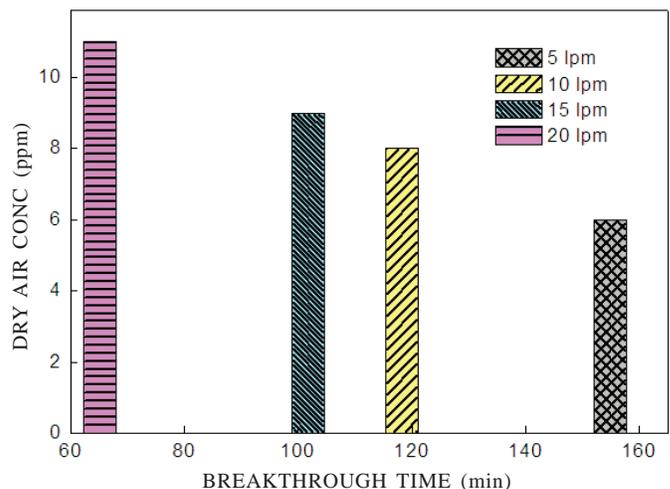

Figure 7. Mes concentrations (5 per cent (v/v) DMMP in MeS experiment) in dry air at Bt.

the major portion of the bed to be free from a contaminant and ready for the cycle to repeat. After each breakthrough experiment, molecular sieves were collected from upper, middle, and the lower portion of each PSA tower. The FT-IR studies were performed by KBr pellet method to confirm the adsorption of MeS and DMMP over 13X. The MeS adsorbed 13X molecular sieves only aromatic ring stretching modes 1620 cm⁻¹ – 1260 cm⁻¹ (Fig. 8(a)) were observed[20]. Whereas,
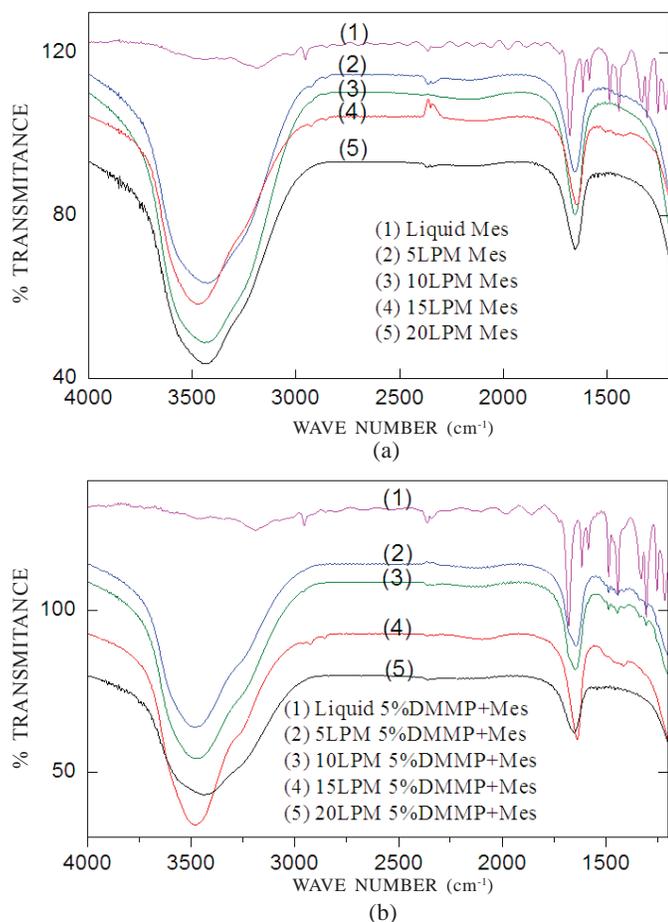
Figure 8. FT-IR of (a) MeS (b) 5 per cent (v/v) of DMMP in MeS adsorbed 13X molecular sieves.

5 per cent(v/v) DMMP in MeS mixture (Fig. 8(b)) the only MeS aromatic ring stretching frequencies were clearly visible and no characteristic peaks of DMMP (1240 cm^{-1}characteristic peak of P = O bond) were noticed for mixture because of low abundance in the mixture[21]. For the sake of comparison, the liquid sample of MeS and 5 per cent (v/v) DMMP in MeS is also provided in Fig. 8. The FT-IR analysis clearly demonstrates that the MeS retains on 13X.

3.4 Regeneration Temperature of 13X

The MeS or DMMP may strongly bind to the surface of the 13X and may not desorb by simple pressure or purge cycle and may retain on the 13X. Therefore, the 13X may warrant either replacement or maintenance cycle after prolonged operation. Hence, TPD of probe molecules gives an idea for temperature required for regeneration of molecular sieves instead of replacement of molecular sieves. TPD experiment of MeS and DMMP were performed over 13X molecular sieves at atmospheric pressure. The MeS and DMMP were adsorbed on 13X (100 mg) individually at 50 °C by injecting the liquid MeS/DMMP into preheated injector using He (100 ml/min) as a carrier. Desorption profile was monitored by increasing the temperature from 30 °C to 500 °C at a rate of heating 8 °C/min. MeS desorption pattern (Fig. 9) revealed multi-step desorption with an onset temperature of 140 °C with a strong desorption

peak centred at 320 °C. DMMP desorption pattern (Fig. 9) revealed a two-step desorption with an onset temperature of 140 °C with a T$_{max}$ at 185 °C (as shoulder peak) for weakly adsorbed DMMP molecules followed by an intense desorption peak centered at 320 °C approximate for strongly adsorbed DMMP molecules. The 13X can be easily regenerated at ~320 °C based on the TPD of MeS and DMMP experiments[21].
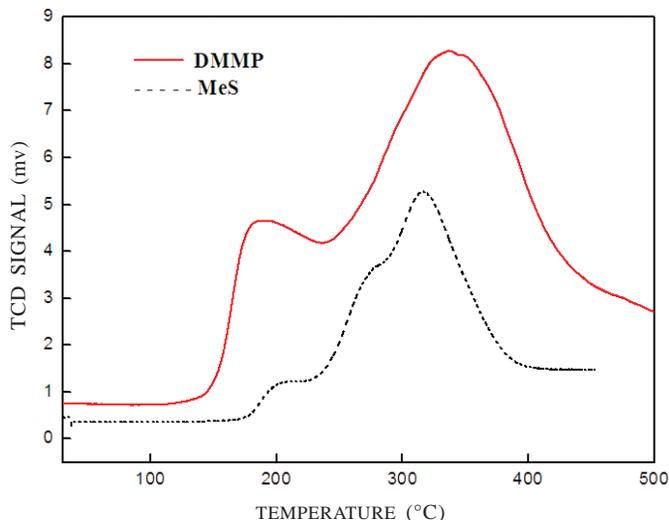

Figure 9. Temperature programmed desorption profile of DMMP and MeS.

4. CONCLUSIONS

In the present study, a PSA test procedure was established for high boiling chemicals such as MeS and DMMP, which are widely accepted as surrogates for the blister and nerve agents using 13X molecular sieves. The sorption and solvent extraction method by XAD-2 are promising for the low volatile compounds, which cannot be analysed and quantified accurately by real-time analytical technique. The influence of feed flow rate is more significant on the breakthrough time. TPD of MeS and DMMP confirm that the relatively polar molecules (DMMP) tend to adsorb strongly compared to that of less polar molecules (MeS). The 13X molecular sieves pore size distribution data reveal that the 8.15Å, is suitable pore size, for the blister and nerve agents. The 13X can easily be regenerated at ~320 °C for maintenance purpose for the PSA after prolonged field operations. The PSA system appears to be the promising technology for air purification applications specific to the CWAs simulants.

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Conflict of Interest: None

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