Effect of precipitation and contamination origin on the efficiency of pinacolyl alcohol identification in concrete debris

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Abstract. **Chemical warfare agents (CWAs) pose a significant threat to people and the environment. Nowadays, the war fights take place mostly in urban areas. Here, chemical weapons contaminate materials of different properties, and the behavior of the parent contaminant may vary. Concrete has an alkaline pH and rapidly decomposes chemical warfare agents. The study deals with the analysis of potentially contaminated concrete samples taken from the site of the alleged use of nerve CWA soman (GD, pinacolyl methylphosphonofluoridate) using gas chromatography. The final degradation product of soman alkaline hydrolysis – pinacolyl alcohol (3,3-dimethylbutan-2-ol) – was chosen as the analyte. The method for the preparation of the concrete samples included organic solvent extraction of the contaminant, in which two organic solvents with different polarity, namely acetone and ethyl acetate, were used separately for comparison. The applicability of the method; the extraction efficiency from concrete debris at given time intervals from the time of contamination to the start of extraction; the effect of moisture addition before and after contamination; and the effect of the extractant used were studied. The possibilities of wipe sampling of the concrete surface in case of point and area contamination with pinacolyl alcohol were also monitored. The precision of the quantitative analysis was expressed by measuring the standard deviation and was worse in the case of ethyl acetate. The highest recovery values were observed with extraction from dry concrete, followed by concrete moistened after contamination. In the case of area contamination, a lower efficiency of surface wipe sampling was found. The results are particularly useful in the field analysis of samples after the use of chemical weapons.**

Keywords: ***Chemical weapons, field analysis, gas chromatography, nerve agents, soman, wipe sampling.***

I. INTRODUCTION

In today's world, conducting military operations in urban areas is becoming much more common than in the past, and in future conflicts this form of operation is inevitable [1]. Chemical warfare agents (CWAs) still pose a significant threat to people and the environment, and the knowledge of the collection and subsequent preparation of a sample plays an important role for the identification of a CWAs by commonly used gas chromatography and mass spectrometry (GC/MS) in military deployable laboratories [2].

Nerve agent soman, (GD; pinacolyl methylphosphonofluoridate) belonging among the Schedule 1 substances of the Chemical Weapons Convention, possesses several noteworthy properties. Its toxicity characteristics include a low lethal dose to humans (LD_{50}), which stands at 0.35 grams [3], [4]. Additionally, GD is resistant to antidotes due to its rapid ageing in the body. Furthermore, it demonstrates relatively high persistence in the environment, with a vapor pressure (VP) of 53.3 Pa at 25°C [4]. GD is colorless to brown liquid that is relatively odorless in its pure state, but impurities may cause a fruity or camphor odor [4]. It is slightly soluble in water (2.1 %) and very soluble in fats [4] and can therefore easily penetrate the human skin [5].

Hydrolysis of GD is a common process of environmental degradation. It occurs across a range of pH environments, including neutral, acidic, and basic conditions. However, alkaline hydrolysis stands out as the most efficient mechanism [5]. Consequently, when decontaminating surfaces contaminated with GD, alkaline hydrolysis serves as the primary method [5]. The intermediate product of GD hydrolysis is pinacolyl methylphosphonic acid, which is further and much more slowly hydrolysed to the final hydrolysis products, methylphosphonic acid and pinacolyl alcohol (PA), (3,3-dimethylbutan-2-ol) [5]. The reaction takes up to 60 h at pH 6 and 25 °C. In diluted solutions, GD is hydrolysed within 1.8 min at pH 10.8 [6] and the reaction rate

increases with temperature [5]. Degradation products can also be used as a longer-term indicator of the use of GD in the environment and may play a key role in the identification of the parent agent [5]. Moreover, PA does not need to be derivatised for identification by gas chromatography, thus providing a good indicator of past GD present [7].

Concrete is an important substrate due to its wide use in construction and constitutes a significant portion of debris generated by a potential attack [8]. Concrete is characterised, among other properties, by porosity, alkaline pH ~ 12 [9] and permeability to liquids, depending on the type of concrete [10]. From this we can conclude that contamination of concrete with liquid GD leads to its penetration into the matrix and subsequent efficient and rapid alkaline hydrolysis to methylphosphonic acid and PA due to high pH.

Identification of Chemical Warfare Agents and their fate in concrete has been the subject of extensive research by various authors. These studies primarily focus on several specific agents, including sulfur mustard [11], [12], sarin [13], tabun [14], adamsite [15] or VX [8]. In the context of preparing samples contaminated with chemical substances related to the Chemical Weapons Convention for analysis "Recommended operating procedures for analysis in the verification of chemical disarmament" (Blue Book) - have been developed by the Finnish Institute for Verification of Compliance with the Chemical Weapons Convention (VERIFIN) including a recommended procedure for the preparation of both concrete. These procedures are validated in OPCW testing and are widely used by laboratories worldwide [16]. The procedure for briefly starts with a small amount of concrete sample (1-5 g), which is homogenized by crushing if necessary. Extraction with an organic solvent (e.g. acetone or dichloromethane), water and 1M HCl are then used in sequence. Finally, the three fractions of the samples are further separated, some of them evaporated to dryness and derivatized with the appropriate reagents if necessary [17]. However, this procedure is time-consuming, difficult to implement and does not suit field conditions. Also, it's worth noting that the influence of meteorological conditions or contamination origin was not thoroughly considered or studied in these investigations.

The aim of the study is to optimize the developed sample preparation method applicable in military deployable laboratories, observe the extraction efficiency of PA from 2 types of concrete debris – lost formwork and steel fibre reinforced concrete (SFRC) at specified periods from the time of contamination to the start of extraction, the effect of moisture addition before and after contamination and the effect of the used extractant – acetone (Acon) and ethyl acetate (Etac). There were also monitored the possibilities of wipe sampling of the concrete surface in case of point and area contamination with PA, simulating accidental spillage of liquid or dispersion from a chemical munition.

II. Materials and methods

A. Reagents and Material

The stock solution (concentration 7.56 mg·mL^{-1}) used for concrete contamination was prepared from 99% pinacolyl alcohol (VOZ Zemianské Kostoľany, Slovakia) dissolved in 99.8% dichloromethane (Merck, Darmstadt, Germany). Ethyl acetate 99.7 % (Sigma-Aldrich, Steinheim, Germany) and 99.5% acetone (Chromservis, Praha-Petrovice, Czech Republic) were used as solvents for the extraction of PA.

The concrete samples consisted of two different types of concrete. The first type was a lost formwork made of plain and lightweight concrete (DITON s.r.o., Stritez, Czech Republic), which represented a less homogeneous and more porous concrete sample. [18]. The second type was cut steel fibre reinforced concrete blocks (dimensions 3.5 cm x 3.5 cm x 3.5 cm, weight 75–85 g) with compressive strength class C30/37 and exposure class XC4 – cyclical wetting and drying [19] this type is used in applications where steel wires completely replace standard reinforcement or are used in combination. A typical example of its use is polished industrial floors [20]. This type of concrete, on the other hand, represented a more homogeneous and less porous concrete sample.

The extracts were analysed by a gas chromatograph with flame ionization detector GC/FID Trace 1310 (Thermo Fisher Scientific Inc., USA). The column was TG-5MS, dimensions 30 m × 0.32 mp × 0.50 m. The column temperature was set so that the temperature was 80 °C for 2 min, then with a temperature gradient of 20 °C · min^{-1} the temperature was increased to 280 °C and then left at this temperature for another 2 min. The total duration of the method was 15 minutes. The injection port temperature was set at 250 °C and the injection was performed in split mode with a ratio of 1:13. A constant flow of 1.5 mL · min^{-1} carrier gas (helium) was applied to the column. The flame ionization detector was used for data acquisition throughout the method, the detector temperature was 280 °C. The flame generation gases were set at flow rates of 350 mL · min^{-1} (air) and 40 mL · min^{-1} (hydrogen). In addition, an additional inert gas (make-up gas) was injected into the system at a flow rate of 30 mL · min^{-1} (nitrogen). A sample volume of 1 μL was injected into the instrument using a TriPlus RSH autosampler (Thermo Fisher Scientific Inc., USA) using the "hot needle" method.

The following instruments were used for sample preparation: ultrasonic bath Sonorex Super RK 106 (Bandelin), laboratory centrifuge Janetzki T5 (LAB system), analytical balance (Mettler Toledo), automatic pipettes (maximum volume 200 μL, 1 mL and 5 mL, Transferpette), laboratory refrigerator and laboratory dryer. Other laboratory equipment included DURAN wide-mouth laboratory bottles with lids (100 mL volume, Fisherbrand), low glass beakers (150 mL volume), hour glass (70 mm diameter), petri dish (60 mm diameter) hammer, tweezers, screw, desiccator, cap vials (5 mL and 2 mL volume).

B. Procedures for monitoring the effect of precipitation

When monitoring the effect of precipitation on extraction efficiency, 3 cases were studied: moisture addition to the sample before contamination, moisture addition to the sample after contamination and dried sample. These cases were monitored for both types of concrete.

1) *Lost formwork samples*

The preparation of lost formwork samples involved breaking the lost formwork with a hammer into concrete fragments weighing approximately 10-30 g, which were then dried in a laboratory dryer (10 min, 100 °C) and then cooled to laboratory temperature in a desiccator.

The moisture addition before contamination involved moistening the concrete fragments evenly with water equal to 3 % of the weight of the concrete fragment before contamination. After 1 minute the fragments were evenly contaminated with 200 µL of 7.56 mg·mL^{-1} PA solution.

The moisture addition after contamination involved even contamination of the fragments with 200 µL of 7.56 mg·mL^{-1} PA solution. After a period of 1 hour, the fragments were evenly moistened with water equal to 3 % of the weight of the concrete fragment.

Contamination of the dried concrete fragments involved even contamination of the fragments with 200 µL of 7.56 mg · mL^{-1} PA.

The samples thus prepared for the 3 different experiments were then left on petri dishes at laboratory conditions for a specified period. Then the samples were transferred to 100 ml wide-mouth Fisherbrand DURAN laboratory bottles, 30 ml of Acon or Etac was applied directly in each bottle and the bottles were tightly capped. These bottles were then sonified in an ultrasonic bath for 30 minutes. After the extraction, 4 mL of the solution was transferred from the bottles into 5 mL vials and centrifuged for 5 minutes. Finally, 1 mL of the cleared solution was transferred into GC vials which were sealed and prepared for the analysis.

2) *SFRC samples*

The preparation of SFRC samples involved drying in a laboratory dryer (10 min, 100 °C) and then cooled to laboratory temperature in a desiccator. Contamination of the SFRC blocks took place only on one side of the block (average area - 10 cm^2).

The moisture addition before contamination involved moistening with 20 µL of water per cm^2 of contamination are. The water droplets were then spread evenly over the area to be contaminated using tweezers, as this type of concrete did not properly absorb liquid. After 1 minute the blocks were evenly contaminated with 100 µL of 7.56 mg·mL^{-1} PA solution.

The moisture addition after contamination involved even contamination of the blocks with 100 µL of 7.56 mg · mL^{-1} PA solution. After a period of 1 hour, the contamination area was moistened with 20 µL of water per cm^2. The water droplets were then spread evenly over the area to be contaminated using tweezers.

Contamination of the dried concrete samples involved even contamination of the one side of the SFRC blocks with 100 µL of 7.56 mg·mL^{-1} PA solution.

The samples thus prepared for the 3 different experiments were then left on petri dishes at laboratory conditions for a specified period. Then the samples were transferred to 150 mL beakers, 10 mL of Acon or Etac was applied at the bottom of each beaker and covered with a petri dish. These beakers were then sonified in an ultrasonic bath for 30 minutes. After the extraction, 2 mL of the solution was transferred and centrifuged for 5 minutes. Finally, 0.6 mL of the cleared solution was transferred into GC vials for analysis.

C. *Procedures for monitoring the effect of contamination origin*

The effect of contamination origin on the extraction efficiency of PA was also monitored for both types of concrete. In this experiment, 100 µL of 7.56 mg·mL^{-1} PA solution for lost formwork samples or 50 µL of 7.56 mg·mL^{-1} PA solution for SFRC samples was applied, depending on the experiment, either to a single point on the sample or the contaminant was applied evenly to cover the largest area. Then, after a specified period, a circular motion was made to wipe using tweezers and cellulose soaked in Acon or Etac. The cellulose was then placed in a 100 mL wide-mouth Fisherbrand DURAN laboratory bottle with 30 mL of Acon or Etac and sonified in an ultrasonic bath for 30 minutes. After the extraction, 4 mL of the solution were centrifuged, and the liquid extract was analyzed.

III. RESULTS AND DISCUSSION

The data processing was carried out based on the determination of 3 calibration series of PA solutions in dichloromethane, a linear calibration curve with a coefficient of determination $R^2 = 0.9991$ was established, which was used to calculate the mass concentrations of PA in the sample. The concentration of the stock solution of PA (7.56 mg·ml^{-1}) was chosen so that the maximum theoretical recovery was within the calibration curve, where the upper point is equal to 50 µg·ml^{-1}. The mass concentration values were converted to the observed extraction efficiency of PA (E_{obs}), i.e. the percentage theoretical recovery of PA throughout the sample preparation. The arithmetic mean of these efficiencies and its standard deviation were calculated from the individual values of the E_{obs} of the sample replicates. The magnitude of the E_{obs} served as a comparative criterion for the different procedures in this work.

A. The effect of precipitation

In this experiment, PA was applied to a) an already moistened concrete sample (moistened before), b) a concrete sample that was moistened 1 hour after contamination (moistened after) and c) a dried concrete sample (dried sample) to compare the resulting extraction efficiency. These cases simulate influence of precipitation and moisture before and after contamination on extraction efficiency and compare extraction efficiency with

Each experiment of concrete lost formwork samples was measured 3 times, in total 174 samples were used, to obtain statistical data and standard deviations for the precision of analysis. The mean values of the detected E_{obs} are shown in the graphs for Acon (Fig. 1.) and Etac (Fig.2.) separately.
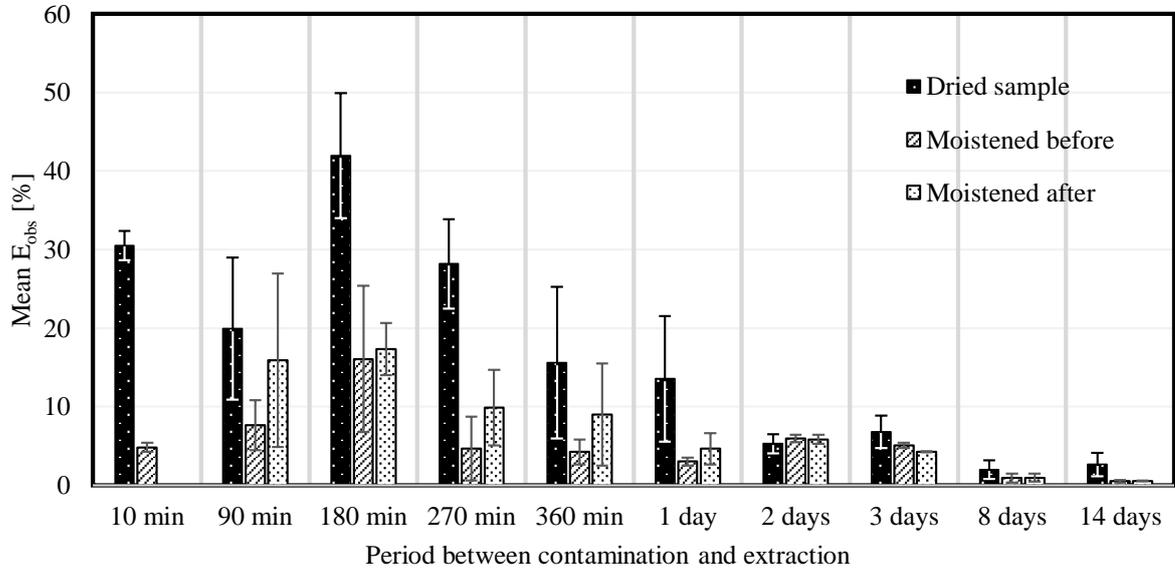
Fig. 1.   The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) at periods using acetone as extractant of concrete lost formwork samples.

contaminated dried samples. The effect of the period from contamination to extraction on E_{obs} was also monitored. A total of 10 periods (10, 90, 270, 360 minutes and 1, 2, 3, 8, 14 days) were tested to monitor were the decrease in concentration within a short time after the contamination and to emphasize the importance of early intervention. The suitability of Acon and Etac used for extraction and both types of concrete samples were also tested and compared.

1) Lost formwork samples
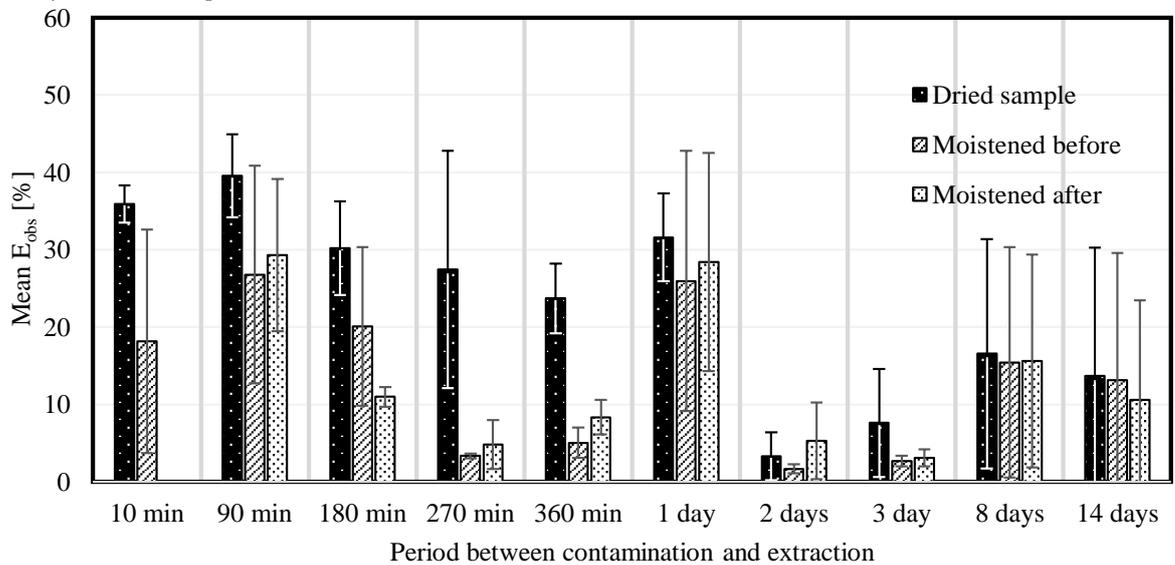
Fig. 2.   The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) at periods using ethyl acetate as extractant of concrete lost formwork samples.

High deviations caused by the different structure of lost formwork fragments were observed. The different structure may have caused volatile PA to evaporate, become trapped deeper in the fragment structure or desorbed with added water. We also observed that E_{obs} is highest in the dried samples where the extraction was not disturbed by the presence of water and lowest when the samples are moistened before contamination where PA could not be absorbed into the concrete to the same extent as in other experiments due to moisture. Etac as an extraction reagent showed higher E_{obs} overall, but also had higher standard deviations then Acon for cases of moistened samples. Acon as an extraction reagent had lower E_{obs} for moistened samples compared to dried samples.

Each experiment of SFRC samples was measured 2 times, in total 116 SFRC blocks were used as samples. The mean values of the detected E_{obs} are shown in the graphs for Acon (Fig. 3.) and Etac (Fig. 4.) separately.

SFRC samples provided overall lower E_{obs} compared to lost formwork samples due to their low porosity and liquid absorption, but compared to the results from the lost formwork, the SFRC samples are more consistent in decreasing E_{obs} over periods due to their more homogenous structure. Etac again showed significantly higher E_{obs} in all 3 cases than Acon. Moistening the samples produced a similar effect to that of lost formwork samples.
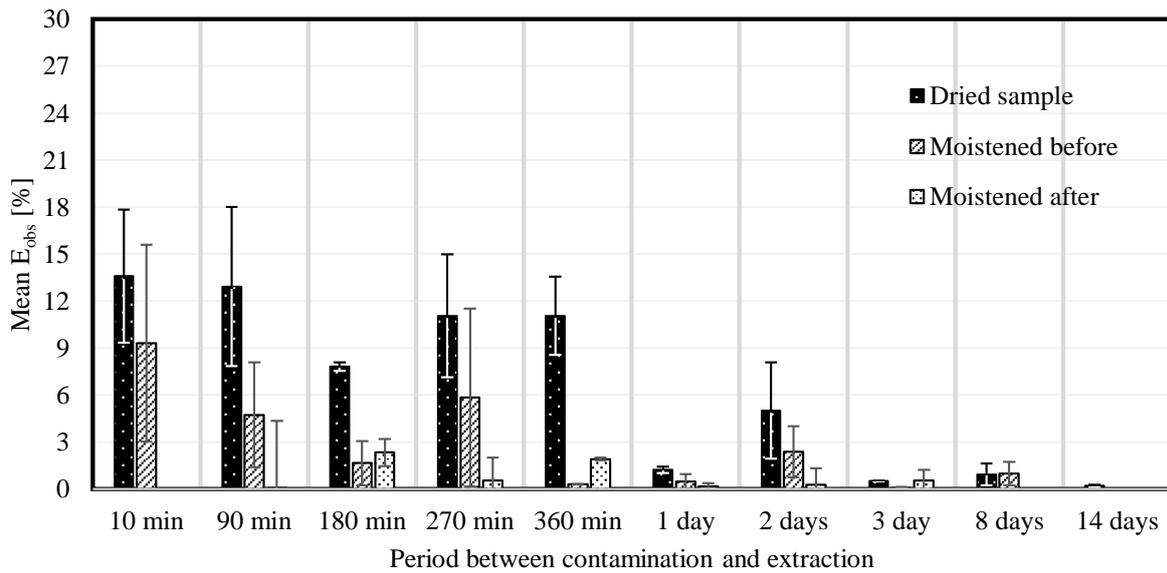
Fig. 3. The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) at periods using acetone as extractant of steel fibre reinforced concrete samples.
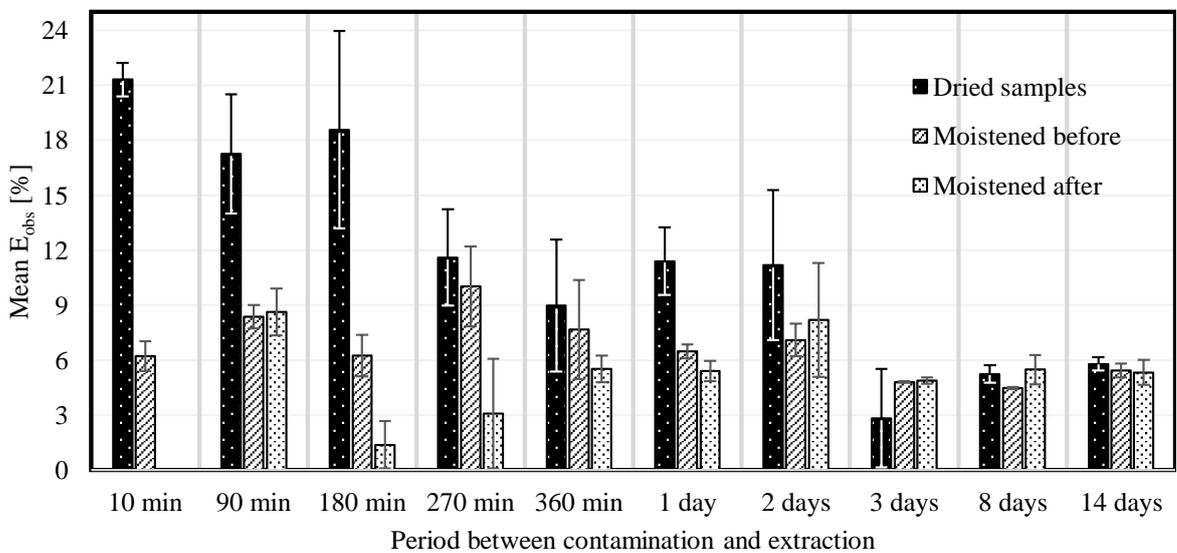
Fig. 4. The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) at periods using ethyl acetate as extractant of steel fibre reinforced concrete samples.

SFRC samples

For SFRC, the lowest results were obtained when analyzing samples that were wetted after contamination. The low porosity of the material causes retention of the contaminant (PA) on the surface. The fate of PA in this scenario is evaporation. When water is added, it washes and dilutes the contaminant from the surface reducing the final sampling recovery. In the case of lost formwork, the porosity is considerably higher. Adding water after contamination does not significantly affect recovery compared to samples moistened before contamination with PA. It can be evaluated that meteorological influences differ for concrete samples based on their technical properties. In summary, understanding these variations is crucial for effective sampling but also decontamination strategies in different concrete scenarios.

B. The effect of contamination origin

In this experiment, the effect of area and point contamination of concrete on extraction efficiency was studied using 2 cases that simulated dispersion of CWA by chemical munitions or other means (area contamination) and accidental spillage of liquid on concrete (point contamination). In the case of area contamination, sample was evenly contaminated with PA and in the case of point contamination, the contaminant was applied to a single point on the concrete sample and after 10 minutes, a wipe was taken using cellulose moistened in used solvent. In this experiment, Acon and Etac solvents were again used as extraction reagents and both types of concretes were used for comparison.

1) Lost formwork samples

Each experiment was measured 5 times to obtain statistical data. The results from the measurement of the
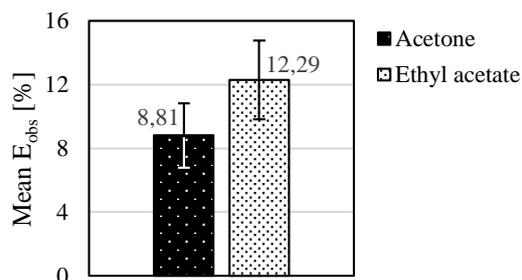
Fig. 5.   The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) for acetone and ethyl acetate as extractant for point contamination of lost concrete

point contamination and area contamination are divided
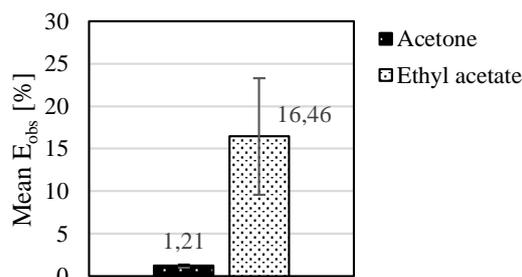
Fig. 7.   The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) for acetone and ethyl acetate as extractant for point contamination of steel fibre reinforced samples

into 2 graphs (Fig. 5., Fig. 6.). E_{obs} of point contamination were generally higher than in the case of area contamination due to a better penetration of PA into the structure of the concrete sample. Also, the E_{obs} for Etac was higher than for Acon.
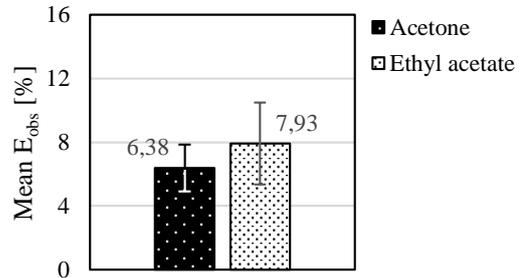
Fig. 6.   The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) for acetone and ethyl acetate as extractant for area contamination of lost concrete
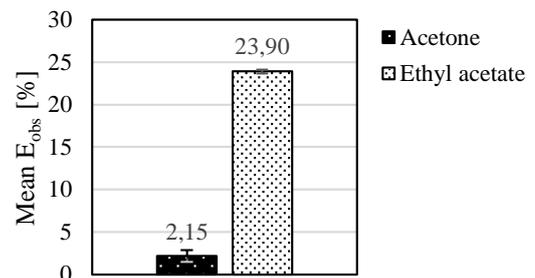
Fig. 8.   The mean values of the observed extraction efficiency of pinacolyl alcohol (E_{obs}) for acetone and ethyl acetate as extractant for area contamination of steel fibre reinforced samples

2) SFRC samples

Each experiment was measured 3 times and the results from the measurement of the point contamination and area contamination are also divided into 2 graphs (Fig. 7., Fig. 8.).

Different results were observed for the SFRC samples than for the lost formwork samples. E_{obs} in the case of point contamination were overall lower than in the case of area contamination due to high evaporation and inability to absorb in less porous material. Etac gave us significantly higher E_{obs} than Acon, which gave E_{obs} within 3 %.

CONCLUSIONS

From the results obtained, we can conclude that precipitation, the origin of contamination, the type of concrete and the extraction reagent have a significant effect on the resulting extraction efficiency of PA.

The highest extraction efficiency values were observed with extraction from dry concrete, followed by concrete moistened after contamination. Acetone had an overall lower extraction efficiency than ethyl acetate and a markedly lower extraction efficiency when the concrete sample was moistened than in case of dried concrete but showed smaller standard deviations. Ethyl acetate had a higher extraction efficiency compared to acetone but is not

very suitable for quantitative analysis due to the standard deviations obtained. Ethyl acetate also produced additional unwanted peaks on the chromatograms which could interfere with the analysis. Therefore, for identification purposes, acetone is a better extraction reagent.

In the case of area contamination, a lower efficiency of surface wipe sampling was found when using samples from porous lost formwork samples, but a higher efficiency of surface wipe sampling was observed when using less porous SFRC samples.

The work is limited by testing in laboratory conditions that do not fully reflect real conditions. Temperature is chosen constant, rainfall is simulated by the addition of water, the effect of wind is omitted. Nevertheless, the results allow us to make assumptions for development in a real environment.

For comparison, the method presented in the Blue Book is more time-consuming and more difficult, which is not suitable for field conditions. The Blue Book also assumes smaller sample volumes and possible homogenization by crushing, which is only possible if a laboratory crusher is used. Homogenization leads to contamination of the instruments used and to loss of contaminant due to evaporation. In addition, due to the high porosity of concrete, wipe sampling appears to be a less effective sampling method and may not lead to successful identification.

In summary, the developed method is a simple but applicable method for the identification of contaminants in concrete samples. The results show that understanding meteorological factors, properties of concrete and their impact on extraction efficiency is crucial for effective contaminant identification on concrete samples and decontamination strategies. Precipitation reduces the analytical recovery, as does a more homogeneous type of concrete. Further research should include the use of the CWAs themselves and other degradation products as contaminants to validate the method.

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REFERENCES

[1] A. Vautravers, "Military operations in urban areas," *International Review of the Red Cross*, vol. 92, no. 878, pp. 437–452, Jun. 2010, doi: https://doi.org/10.1017/s1816383110000366.

[2] J. T. Kelly, A. Qualley, G. T. Hughes, M. H. Rubenstein, T. A. Malloy, and T. Piatkowski, "Improving Quantification of tabun, sarin, soman, cyclosarin, and sulfur mustard by focusing agents: A field portable gas chromatography-mass spectrometry study," *Journal of Chromatography A*, vol. 1636, p. 461784, Jan. 2021, doi: https://doi.org/10.1016/j.chroma.2020.461784.

[3] "Schedule 1," *OPCW*. https://www.opcw.org/chemical-weapons-convention/annexes/annex-chemicals/schedule-1

[4] D. Hank Ellison, *Handbook of Chemical and Biological Warfare Agents, Volume 1*. 2022. doi: https://doi.org/10.4324/9781003230571.

[5] I. S. Che Sulaiman *et al.*, "A review on analysis methods for nerve agent hydrolysis products," *Forensic Toxicology*, vol. 38, no. 2, pp. 297–313, Dec. 2019, doi: https://doi.org/10.1007/s11419-019-00513-x.

[6] S. Popiel and M. Sankowska, "Determination of chemical warfare agents and related compounds in environmental samples by solid-phase microextraction with gas chromatography," *Journal of Chromatography A*, vol. 1218, no. 47, pp. 8457–8479, Nov. 2011, doi: https://doi.org/10.1016/j.chroma.2011.09.066.

[7] T. Rozsypal, Kristyna Zitova, and Ludmila Mravcova, "Overcoming the BSTFA: Study on Trimethylsilylation Derivatization Procedures for Chemical Weapons Convention-Related Alcohols in Field Analysis," *Analytical Letters*, pp. 1–17, Nov. 2023, doi: https://doi.org/10.1080/00032719.2023.2281587.

[8] J. W. Williams *et al.*, "Degradation Kinetics of VX on Concrete by Secondary Ion Mass Spectrometry," *Langmuir*, vol. 21, no. 6, pp. 2386–2390, Feb. 2005, doi: https://doi.org/10.1021/la047933j.

[9] Y. Sumra, S. Payam, and I. Zainah, "The pH of Cement-based Materials: A Review," *Journal of Wuhan University of Technology-Mater. Sci. Ed.*, vol. 35, no. 5, pp. 908–924, Oct. 2020, doi: https://doi.org/10.1007/s11595-020-2337-y.

[10] A. M. Neville, *Properties of concrete*. Harlow Pearson Education, 2012.

[11] C. A. S. Brevett, K. B. Sumpter, G. W. Wagner, and J. S. Rice, "Degradation of the blister agent sulfur mustard, bis(2-chloroethyl) sulfide, on concrete," *Journal of Hazardous Materials*, vol. 140, no. 1–2, pp. 353–360, Feb. 2007, doi: https://doi.org/10.1016/j.jhazmat.2006.09.067.

[12] H. Jung and S. Choi, "Behavior of sulfur mustard in sand, concrete, and asphalt matrices: Evaporation, degradation, and decontamination," *Journal of Environmental Science And Health, Part A*, vol. 52, no. 12, pp. 1121–1125, Jul. 2017, doi: https://doi.org/10.1080/10934529.2017.1342498.

[13] C. J. O'Brien, J. A. Greathouse, and C. M. Tenney, "Dissociation of Sarin on a Cement Analogue Surface: Effects of Humidity and Confined Geometry," *The Journal of Physical Chemistry C*, vol. 120, no. 49, pp. 28100–28109, Dec. 2016, doi: https://doi.org/10.1021/acs.jpcc.6b10046.

[14] H. Jung, J. Lee, H. Park, and J. A. Seo, "Fate of Nerve Agent Tabun in Concrete and Soil: Evaporation and Decontamina-tion," *Environmental Engineering Science*, vol. 36, no. 6, pp. 650–655, Jun. 2019, doi: https://doi.org/10.1089/ees.2018.0535.

[15] T. Rozsypal, "Use of aliphatic thiols for on-site derivatization and gas chromatographic identification of Adamsite," *Journal of The Serbian Chemical Society*, vol. 88, no. 6, pp. 639–652, Jan. 2023, doi: https://doi.org/10.2298/jsc221207025r.

[16] "Blue Book | VERIFIN | University of Helsinki," *www.helsinki.fi*. https://www.helsinki.fi/en/verifin/about-verifin/blue-book

[17] M. L. Kuitunen and N. Hamzah, *"Concrete samples"* in P. Vanninen (ed.) *"Recommended operating procedures for analysis in the verification of chemical disarmament, 2023 Edition"*, University of Helsinki, Finland, 2023, pp. 335–342. http://www.helsinki.fi/verifin/bluebook

[18] Precast concrete products – Normal weight and lightweight concrete shuttering, EN 15435, 2008.

[19] Concrete – Specification, performance, production and conformity, EN 206+A2, 2013.

[20] T. Błaszczyński and M. Przybylska-Fałek, "Steel Fibre Reinforced Concrete as a Structural Material," *Procedia Engineering*, vol. 122, pp. 282–289, 2015, doi: https://doi.org/10.1016/j.proeng.2015.10.037.