

The Potential of Agro Based Nanomaterials for Nanofilters to Capture Suspended Titanium Nanoparticles in the Air

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ABSTRACT : *Nanomaterials have a wide range of new technologies and industrial use and have created many new products and employment opportunities. However, they can also pose unknown risks and specific uncertainties in occupational safety and health issues. The latest and most worrying issue involves the increasing production and nanoparticles, particularly titanium dioxide (TiO₂). Therefore, a rigorous study should be carried out to obtain more intensive information to develop a new technique for personal exposure monitoring. The commercially available nanoparticle respiratory deposition (NRD) sampler usually occupied with nylon filter contains TiO₂ background material and is rather expensive. As an alternative, agro based nanofilters were developed from nanomaterials synthesized from rice husks, namely, nanosilica and nanozeolite embedded on/in a polyvinylidene fluoride (PVDF) membrane. As a comparison, graphene was also used to produce nanofilters due to its outstanding performance in chemical absorption. Analysis using Field Emission Scanning Electron Microscope (FESEM) showed a formation of cracks on both nanofilters when embedded with 1% w/v of either nanosilica and nanozeolite compared to 0.1 and 0.5 % w/v. Agglomerate of nanosilica particles with the size between 20 – 40 nm and nanozeolite with the size between 18 – 30 nm were identified on the developed nanofilter. Energy Dispersive X-ray (EDX) confirmed the presence of functional groups such as silica, oxide, sodium, alumina, and carbon on the developed nanofilters, further confirming the deposition of the nanomaterials on the PVDF membrane. Further investigation on the ability to capture titanium nanoparticles using 0.1 % w/v nanofilters from both materials showed that all filters tested could capture titanium nanoparticles with nanozeolite filters showing the highest accumulation with 9170 mg/m³. These results suggest that agro-based nanomaterials can be used as nanofilters to capture titanium nanoparticles in the air.*

Keywords - *Exposure Assessment, Nanofilter, Nanoparticle, Nylon, Personal Sampler, Titanium Dioxide*

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1.0 INTRODUCTION

Exposure of the nanoparticle to workers and consumers has become a problem. TiO₂ is one of the substances which has been used excessively as a nanoparticle in many industries. TiO₂ is a chemically inert, semiconducting material. TiO₂ also exhibits photocatalytic activity in the presence of light with an energy equal to or higher than its band-gap energy. The increasing production of nano-sized TiO₂ powder has led to growing concerns about the consequences of exposure to workers' health. According to the National Institute of Occupational Safety and Health (NIOSH), the respirable dust concentration of TiO₂ was 0.10–0.141 mg/m³. In comparison, the time-weighted average (10-h TWA) concentrations of TiO₂ was 2.4 mg/m³ for fine and 0.3 mg/m³ for ultrafine (Current Intelligence Bulletin 63, 2011). Despite the regulation that has been regulated, many cases reported that some industries already exceeded the permissible limit.

Consequently, some of the studies have shown the effect of TiO₂ on human health, such as oxidative stress, genotoxicity, immunotoxicity, and dermal exposure. For example, Brun et al. (2014) demonstrated no visible dissolution of TiO₂ particles for as long as 24 hours after the uptake by human gut epithelial cells grown in vitro monocultures. The result shows, the cytotoxic effect caused by TiO₂ is more due to its size than the metallic ions being released from the particles absorbed by the cells. Due to the size, the nanoparticle is considered more toxic than the larger particles of the same composition. On the other hand, the study done by Lin Zhao et al. (2016) adopted off-line filter-based sampling combined with real-time activity-based monitoring to measure the concentrations in a selected workplace manufacturing workshop in China. This study found that mass concentrations of TiO₂ were at a relatively low level in the packaging workshop (total TiO₂: 46.4 µg/m³, nano TiO₂: 16.7 µg/m³) and milling workshop (total TiO₂: 39.4 µg/m³, nano TiO₂: 19.4 µg/m³) by ICP-MS. The number concentration, surface area concentration of aerosol particles potentially deposited in alveolar, and tracheobronchial regions of lungs in the packaging workshop were $(1.04 \pm 0.89) \times 10^5$ particles/cm³, 414.49 ± 395.07 , and 86.01 ± 83.18 µm²/cm³, respectively, which were all significantly higher than those of the milling workshop [$(0.12 \pm 0.40) \times 10^5$ particles/cm³, 75.38 ± 45.23 , and 17.60 ± 9.22 µm²/cm³, respectively] as well as in the executive office and outdoor background ($p < 0.05$). Activity-related characteristics were found in both workshops, and the time-variant characteristics showed very similar trends for three days in the packaging workshop.

The available nanoparticle personal sampler, a Nanoparticle Respiratory Deposition (NRD) sampler, as shown in Fig. 1, has some limitations towards analyzing the nanoparticles metal oxide such as TiO₂. The nylon filter used in the NRD sampler was discovered to contain titanium on the background level. A higher concentration of the element was detected during the quantitative analysis (Cena et al., 2011 and Mines et al., 2016). This would directly impact the actual measurement of the exposure level of the TiO₂ nanoparticles during the air sampling. It also highlighted that each session of the air sampling required eight nylon filters to be used in the sampler, which is very costly and its limitation suitability for high air temperatures and humidity in the employment sectors in Malaysia.

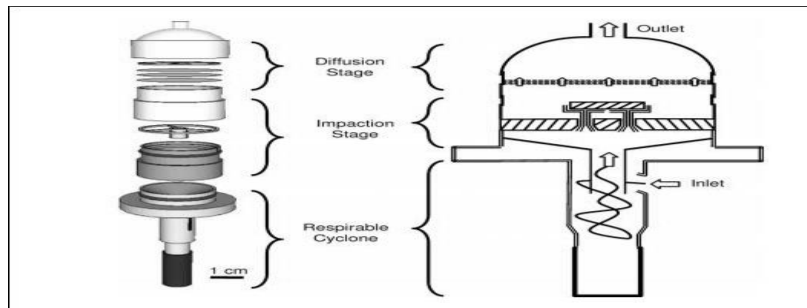


Figure 1 The Components and Schematic Drawing with Airflow Paths of the NRD Sampler (Cena et al., 2011).

Rice husk is an agro-waste material that can form different nanomaterials such as nanosilica and nanozeolite. Nanosilica has potentially great adsorptive material due to its promising properties, i.e., having a large surface area and porous structure (Liou & Yang, 2011). Furthermore, silica particles are also being used to isolate particulate matter fractions in other applications (Stillwell, 2016). Nanozeolite are nano-size hydrated porous crystalline aluminosilicates with an open-framework structure made up of tetrahedral SiO_4^{4-} and AlO_4^{3-} units capable of metal and ion-exchange activities (Ng et al., 2015). In this process, both nanosilica and nanozeolite were used as a precursor to developing nanofilters to capture titanium nanoparticles in the air. In addition, due to its outstanding performance in chemical adsorption, graphite in the form of graphene oxide was also used to produce nanofilter to compare its performance with the agro based nanofilters.

2.0 METHOD

The production of the nanomaterials was using rice husk ash and graphite as the precursors. As a part of previous work by Zuraiddi et al., An experimental setup as shown in Fig. 2, the rice husk ash was used to prepare nanozeolite and nanosilica. In contrast, graphite was used to produce graphene oxide (GO). Three different concentrations of nanomaterials using nanosilica, nanozeolite, and graphene (0.1% w/v, 0.5% w/v & 1% w/v) were used to develop the nanofilter by using the layer deposition method that is facilitated by vacuum filtration adapted from Wang et al. (2019). The filters were produced in three steps. Firstly, five ml from each nanosilica solution were pipetted on PVDF membranes (0.22 μm). Then, the solutions were vacuum filtered to produce a PVDF-nanosilica filter. Lastly, the filters were left to dry at room temperature. These steps were repeated until eight filters from each concentration were made.

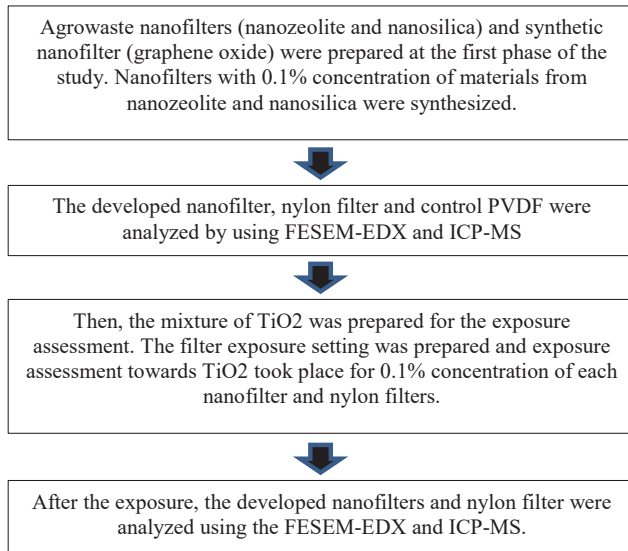


Figure 2 Overall Flowchart of the Study Methodology

The developed PVDF-nanofilters were imaged through Field-Electron Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (FESEM-EDX – Jeol Ltd. Manufacturer, Japan) at University Malaysia Pahang (UMP). Sample preparation has been conducted before analysis. The raw PVDF filter and PVDF-nanofilters (0.1%, 0.5% & 1% w/v concentration) were cut and pasted on a metal plate using a carbon tape. Then, the sample was coated with platinum and exposed to an emitted electron beam.

The filters were placed inside the NRD sampler. The TiO₂ nanoparticles were exposed to the filter using the air pump force to test the performance of nanofilters and capture titanium nanoparticles. Nanofilters were placed carefully on the last cassette (third cassette) of the NRD sampler with the side of the implemented filter against the airflow to collect the nanoparticles, as shown in Fig. 3 (a & b). All sampler components were correctly placed at the neck of the Buchner flask before sealing them properly with masking tape and parafilm. An air tube was connected from the outlet of the sampler to the vacuum pump. It will draw the air from inside the flask. Meanwhile, another tube connected from the sidearm tube of the flask to the flow rate controller, which connected to the air pump as well.

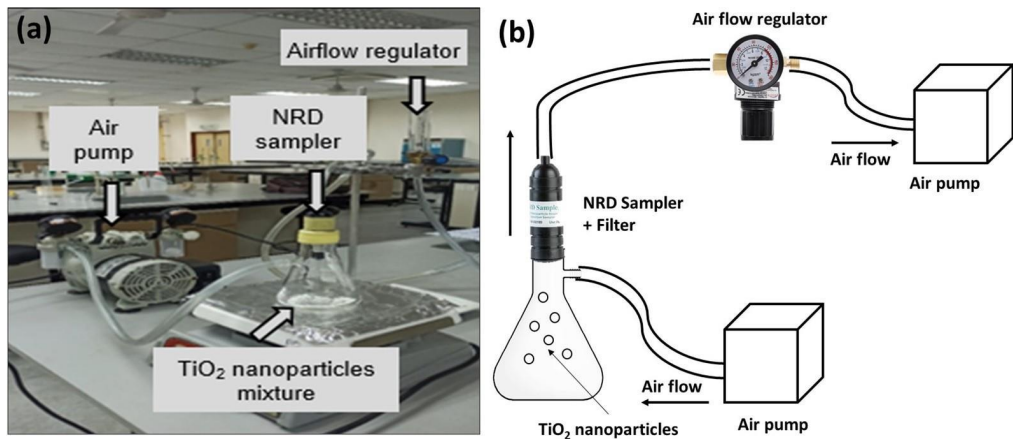


Figure 3 Overall Set Up for in Situ Exposure Assessment

The flow rate controller was adjusted to 2.5 Liters Per Minute (LPM), which followed the American Conference of Governmental Industrial Hygienists (ACGIH) after switching the vacuum pump and air pump. The exposure testing was conducted for 15 minutes. The flask was gently shaken after five minutes-interval to distribute the nanoparticle mixture simultaneously after 15 minutes before all the air tubes connected to the sampler, and the flask was detached completely.

The nanofilter was removed carefully from the cassette before being placed in a clean, dry petri dish. The petri dish was sealed correctly with parafilm to prevent the nanofilter from being contaminated. The petri dish was also labeled correctly based on the type of nanofilter and its concentration. The experiment continued by testing the nylon filter, PVDF membrane, and other new nanofilter already synthesized before. A triplicate sample for each type of nanofilters was submitted to UMP central lab to be quantified using an Inductive Coupled Plasma Mass Spectrometer (ICP-MS) for the presence of captured titanium element. The *in-situ* exposure assessment experiment was set up in the laboratory with precautions measures to reduce the risk of inhalation of TiO_2 .

3.0 RESULTS AND DISCUSSION

Pre-analysis of nanofilters (before exposure to TiO_2) was done using FESEM-EDX to analyze the distribution and stability of each nanomaterial embedded on the PVDF membrane using the layer deposition method. Fig. 3 a) illustrates the pristine PVDF filter with intertwined fibers forming a porous network with a size around $0.3 - 0.6 \mu\text{m}$. However, the porous network was successfully covered with nanosilica when deposited with 0.1, 0.5, and 1.0 % w/v nanosilica (Fig 4 b, c, and d). The nanosilica deposited on the surface of PVDF was seen to be in the form of agglomerates. Each nanoparticle is in the range of $20 - 40 \text{ nm}$. Fair distribution of silica on PVDF-silica composite membrane is also observed in other studies. Fernandes et al. (2017) utilized the immersion precipitation method, while Xiong et al. (2013) used the electrospinning method to prepare the composite filter for wastewater remediation. The good dispersion and distribution of silica nanoparticles suggest the interaction between the PVDF chains and the silanol groups of silica (Zhang, 2011). It is expected that incorporating silica into the PVDF membrane would improve PVDF surface structure by forming more pores and increase the efficiency of the composite filter in trapping contaminants (Fernandes, 2017). A similar process was observed when 0.1, 0.5, and 1.0 % w/v of nanozeolite was deposited on the PVDF membrane (Fig. 5 b, c, and d). Each nanozeolite particle embedded on the PVDF membrane was observed to be in the range of $18 - 30 \text{ nm}$. Recent work has shown that the surface modification of the PVDF membrane by nanozeolite could increase its porosity but did not compromise its permeability (Nassrullah et al. 2020). The modification of the hydrophilic

PVDF membrane with hydrophilic nanomaterials also increases the polarity on the surface, thus increasing the tendency for inorganic nanomaterials such as TiO₂ to bind to the surface. The FESEM image of the GO-PVDF membranes indicates the presence of graphene oxide layering on top of the porous PVDF membrane surface based on the presence of a thin and wrinkle-like layer of the GO. The presence of wrinkle-like GO layering on the PVDF membrane increased with the increased amount of GO (Fig. 6 b, c, and d). The higher the concentration of GO used, the appearance of crumple and wrinkle structures become more accentuated on the surface of the composite filter prepared, which indicated thicker GO sheets were stacked on the surface.

EDX results of pristine PVDF membrane revealed the presence of high carbon (C) content followed by fluorine (F) and small amounts of oxygen (O) (Fig. 6 a). Meanwhile, nanosilica filters showed the presence of major elements in nanosilica, such as silica (Si) and oxygen (O), with a small concentration of carbon (C) impurities present (Fig. 7 b). As for nanozeolite, the presence of the leading chemical composition of the nanozeolite was observed, such as silica (Si), alumina (Al), oxide (O), and sodium (Na) (Fig. 6 c). The sodium present in this analysis is suggested to reduce the acidity of the zeolite acid sites (Saeed, Hamadi, and Sherhanm, 2019). As for the PVDF membrane embedded with GO, it was found that the weight of % of carbon and fluorine were higher in the control PVDF membrane as PVDF is a thermoplastic fluoropolymer with carbon-fluorine and hydrogen elongated chain. The presence of a high % of oxygen element indicates the successful oxidation and graphene oxide formation. In addition, the fluorine weight % was significantly reduced compared to the control PVDF membrane, which is also attributed to the increased thickness of GO that layered the PVDF membrane. In terms of nanomaterials film stability on the filter substrate, it can be observed that a high concentration of nanomaterials with 1% w/v showed an unstable deposition on the filter substrate. This is due to the formation of cracks on the nanoparticle layer (Fig. 8). It could be due to the excessive amount of nanomaterials added onto the membrane, which could cause leakage to the filter if it is being applied. The pressure caused by the airflow could also easily break the nanomaterials layer, affecting the accumulation of the titanium nanoparticles captured. Due to this, filters containing 0.1 % w/v of nanomaterials only were chosen to be tested following its stable deposition on the PVDF membrane.

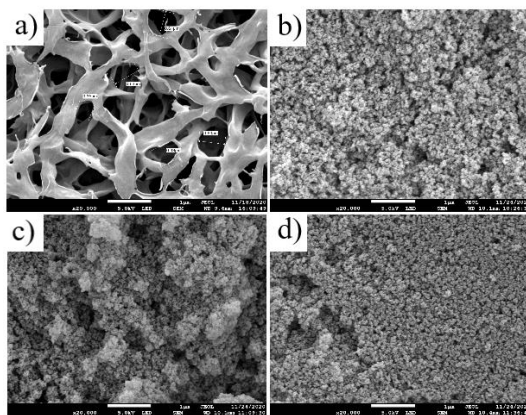


Figure 4 FESEM Images of Nanosilica Embedded on PVDF Membrane before Exposure to Titanium Under 20,000 Magnification A) PVDF Only B) 0.1% W/V Nanosilica - PVDF C) 0.5% W/V Nanosilica - PVDF D) 1.0% W/V Nanosilica - PVDF

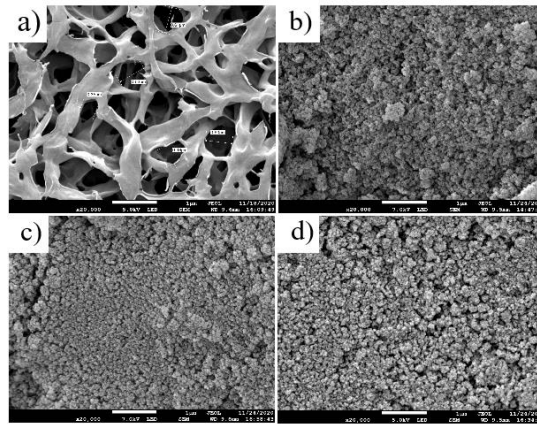


Figure 5 FESEM Images of Nanozeolite Embedded on PVDF Membrane before Exposure to Titanium Under 20,000 Magnification A) PVDF Only B) 0.1% W/V Nanozeolite - PVDF C) 0.5% W/V Nanozeolite - PVDF D) 1.0% W/V Nanozeolite – PVDF

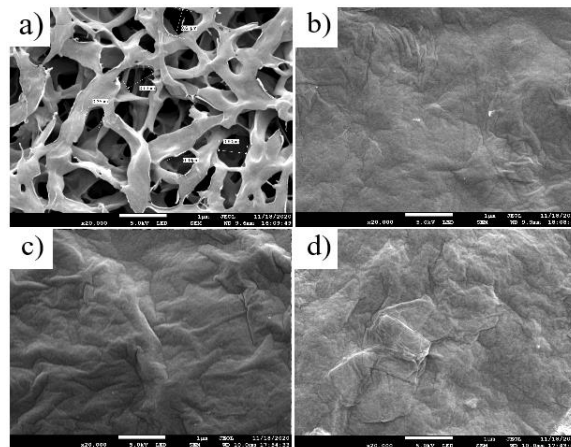


Figure 6 FESEM Images of Graphene Oxide Embedded on PVDF Membrane before Exposure to Titanium Under 20,000 Magnification A) PVDF Only B) 0.1% W/V Graphene Oxide - PVDF C) 0.5% W/V Graphene Oxide – PVDF D) 1.0% W/V Graphene Oxide – PVDF

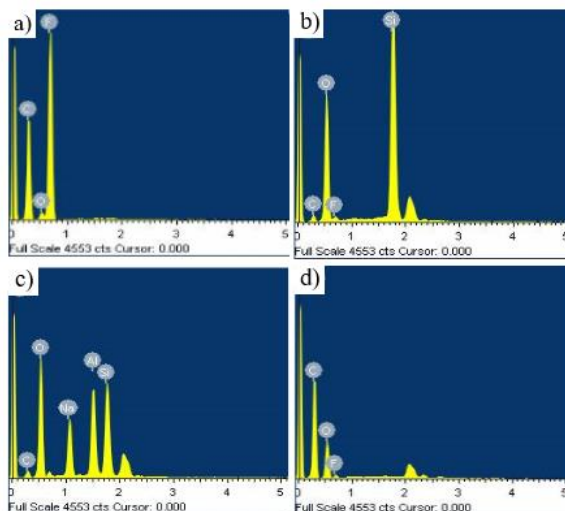


Figure 7 EDX Spectrum for A) PVDF B) Nanosilica - PVDF C) Nanozeolite - PVDF D) Graphene Oxide – PVDF

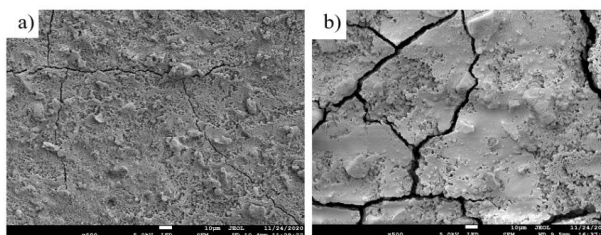


Figure 8 FESEM Images at 500x Magnification of A) 1.0% W/V Nanosilica - PVDF and B) 1.0% W/V Nanozeolite – PVDF Showing the Formation of Cracks on the Surface of the Membrane

From the ICPMS analysis, the result showed that nylon filters have the highest amount of TiO_2 . The average amount of TiO_2 between the triplicate samples for nylon filters is 1326000 mg/m^3 . However, this may be counted as invalid due to its composition of TiO_2 , and each of the nylon filters may have a different concentration of TiO_2 (Mines et al., 2016). Interestingly, nanozeolite-PVDF has the highest amount of TiO_2 trapped among the developed nanofilter with an average of 9170 mg/m^3 , followed by nanosilica-PVDF with 4590 mg/m^3 and GO-PVDF with 2330 mg/m^3 , respectively, as shown in Table 1. On the other hand, the ability of GO-PVDF to trap the TiO_2 during exposure assessment was lower compared to the nanosilica-PVDF and the nanozeolite PVDF. This is due to the limited pores on the GO-PVDF. In addition, the trapping ability of GO-PVDF was not good as compared to others as the GO-PVDF might be lacking cavities with limited pores. However, GO-PVDF was still able to trap TiO_2 based on the ICP-MS analysis. Nanosilica-PVDF was the second-best material to capture TiO_2 .

In contrast to GO-PVDF, the nanosilica-PVDF has a large surface area and high porosity based on the FESEM image. This may also be due to the chemical interaction, such as the bonding between Ti-O-Ti, Ti-O-H, and Ti-O-Si. Other studies stated that chemical bonding occurred between the Si and Ti (Rasalingam et al., 2013; Dalod et al., 2017; Hakki et al., 2017). Other than that, dipole-dipole interaction between the negative charge of the silica and Ti may be contributed to the trapping ability of nanosilica-PVDF towards TiO₂. The best material for capturing TiO₂ is nanozeolite-PVDF, as stated in the result of ICP-MS. Nanozeolite, cheap and agro-waste made, shows a higher captured amount of TiO₂ than nanosilica and graphene oxide. Besides its large surface area and the high porosity, interaction between zeolite and the Ti through the bonding of Ti-O-Na may also contribute to the trapping ability of TiO₂ (Kostrikin et al., 2017).

Table 1 ICP-MS Results on Ti Content from Pre and Post (Before and After Exposure to TiO₂) Filters Exposed to TiO₂. Results are Mean from Triplicates ± SEM.

Sample name	Pre-exposure Results (mg/m ³)	Post-exposure Results (mg/m ³)
PVDF	Not detected	Not detected
Nano silica	Not detected	4586 ± 419
Nano zeolite	Not detected	9170 ± 1123
Graphene	Not detected	2333 ± 239
Nylon	1212000	1326000 ± 28360

4.0 CONCLUSION

Newly developed nanofilters, consisting of 0.1% concentration (nanosilica-PVDF, nanozeolite-PVDF, and GO-PVDF) and nylon filter, were successfully tested after the exposure assessment towards TiO₂ under 15 minutes with a flow rate of 2.5 LPM. The developed nanofilters were tested in the setup of in-situ experiment using the NRD sampler. All the developed nanofilters and the nylon filters managed to capture TiO₂. Although the result showed that the nylon filter has the highest concentration of TiO₂, it is invalid to claim the nylon filter is the best as it already contained high background readings of TiO₂, which affected the result of occupational exposure TiO₂ due to its composition of TiO₂. Moreover, the background reading in the nylon filter already exceeded the recommended exposure limit (REL) of ultrafine TiO₂, which is 0.3 mg/m³. Nanozeolite-PVDF was the best nanofilter as it captured the highest concentration of TiO₂ followed by nanosilica-PVDF and GO-PVDF, respectively. This showed that nanozeolite could potentially replace the nylon filters used in the NRD sampler. The objectives of this study were achieved. However, the exposure assessment method could be improved to test the actual condition of the industry setup. The usage of tape and parafilm to cover the air holes was not so practical. A good layout of the experiment can be done to make the exposure assessment more feasible. For the sample preparation of the ICP-MS analysis, the acid digestion was not fully digested. Suggestions to improve to have different concentrations and types of acid could be helpful to achieve complete digestion of the sample. This could achieve full recovery of TiO₂ from the tested nanofilters.

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