Nature Environment and Pollution Technology
An International Quarterly Scientific Journal
p-ISSN: 0972-6268
(Print copies up to 2016)
e-ISSN: 2395-3454
Vol. 22 | No. 4 | pp. 2189-2194 | 2023
Original Research Paper
https://doi.org/10.46488/NEPT.2023.v22i04.045
Open Access Journal

Assessment of Particulate and Gaseous Fluoride in Phosphate Fertilizer Industry

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Nat. Env. & Poll. Tech.
Website: www.neptjournal.com

Received: 10-03-2023
Revised: 20-04-2023
Accepted: 25-04-2023

Key Words:
Particulate fluoride
Gaseous fluoride
Phosphate fertilizer industry
Respirable dust
Workplace exposure

ABSTRACT

Fluorides are emitted in both gaseous and particle forms in the industrial sector. However, studies usually only report total fluoride content. Therefore, the study aimed to assess the particulate, gaseous fluoride and correlate it with the respirable dust particles in Single Super Phosphate (SSP), Granular Single Super Phosphate (GSSP), and administration divisions of the industry. Respirable dust particles, particulate fluoride, and hydrogen fluoride in the work environment were collected on a filter cassette containing an MCE filter paper (0.8 micron 37-mm) and Na_2CO_3 impregnated backup pad, respectively, using a personal sampler. The fluoride samples were analyzed using Ion Selective Electrode (ISE) and expressed as milligrams per meter cube ($mg.m^{-3}$). The respirable dust, particulate, and gaseous fluoride content were found to have statistically significant differences ($p<0.001$) between the divisions (SSP, GSSP, and administration) in the static monitoring, whereas, in the case of personal monitoring, no significant differences were observed. Average airborne respirable, particulate, and gaseous fluoride levels in static monitoring were 1.37, 1.03, 0.20 $mg.m^{-3}$, 0.018, 0.008, 0.001 $mg.m^{-3}$, and 0.808, 0.403, 0.026 ppm in SSP, GSSP and administration respectively, whereas in personal monitoring the average respirable, particulate and gaseous fluoride concentrations were 1.18, 0.85, 0.30 $mg.m^{-3}$, 0.0013, 0.007, 0.002 $mg.m^{-3}$ and 0.356, 0.258, 0.011 ppm in SSP, GSSP and administration respectively. The present study observed that the levels of fluoride decreased with an increase in distance from SSP, followed by GSSP and administration. It indicates that the fluoride exposure was inversely proportional to the distance of the source. This study outcome will help to design a policy and intervention to mitigate fluoride exposure among workers.

INTRODUCTION

Fluorine (F) is the most electronegative and reactive non-metal element. Therefore, the elemental form of fluorine never occurs in nature, and fluoride pollution mostly occurs through anthropogenic activities in the industrial sector (Jawahar et al. 2022, Wang et al.2019, Sunitha et al. 2022, Torres-Sánchez et al. 2020, Cui et al. 2021). In the fertilizer industry, phosphate fertilizers are produced by adding sulphuric acid to digest rock phosphate, which is necessary to maintain agricultural production worldwide (Millán-Becerro et al. 2022). During this industrial process, particulate and gaseous fluorides are generated. In 2012, hydrofluoric acid (HF) spilled from a chemical plant in Gumi City, South Korea. As a result, many people were injured, and five workers died on-site due to exposure to hydrofluoric acid (Jung & Park 2016, Lee et al. 2016). The exposure to hazardous chemicals in the working environment causes adverse health effects to the workers. Therefore, the workers should be aware of the preventive measures to control the accidents.

Anthropogenic activities, fossil fuel combustion processes, manufacturing operations, and biogenic sources can contribute to the ambient $PM_{2.5}$ particles. In addition to primary released particles, ambient $PM_{2.5}$ contains secondary inorganic particulate fluoride and organic aerosols (Anastasopolos et al. 2022, Fadel et al. 2021). According to Pawar et al. (2014), the mutagenic nature of F was revealed as an increase in the chromosomal aberrations (CA) and sister chromatid exchanges (SCE) frequencies in the lymphocytes of the subjects. Meng & Zhang (1997) showed that the workers chemically exposed to the fumes containing fluoride in the phosphate fertilizer industry could associated with increased Chromosomal aberrations (CA) and micronuclei (MN) frequencies in their blood lymphocytes. Among welders, the relationship between welding fume exposure and respiratory symptoms appears more influenced by fluorides than other particles (Sjögren 2004). Ma et al. (2009) found that the concentrations of serum osteocalcin (OCN) and calcitonin (CTN) increased simultaneously in a Fluoride-exposed worker population.

The SSP and GSSP are the major units in phosphate fertilizer production. In the SSP plant, the single super phosphate is produced by the addition of sulfuric acid to ground or pulverized rock phosphate (insoluble in water) to produce single super phosphate (SSP) with a phosphorus content of 16-21% as phosphorus pentoxide (soluble in water). This process involves mixing the sulphuric acid and the rock phosphate in a reactor. The mixture goes onto a moving conveyor in a den, and the semisolid mixture produced from the den is termed a single super phosphate, which is allowed to cure for 10 to 20 days. After curing the product, the cured SSP is taken further to produce a granulated form of single super phosphate (GSSP) in the granulation circuit of the GSSP plant. The fumes containing silicon tetrafluoride (SiF_4) and hydrogen fluoride (HF) gasses generated from the reactor (mixer) and den. Workers' exposure to these fumes containing fluoride compounds released during the industry's processing and handling of fertilizer might be through inhalation or dermal contact. These fumes might pose adverse health effects to the workers working in the industry. We know that no literature is available regarding assessing particulate and gaseous fluoride levels in the phosphate fertilizer industry. Therefore, this study aimed to assess the particulate and gaseous fluoride levels in the working environment of the phosphate fertilizer industry. This study will be useful to evaluate the key sources of the pollutants in various work environments of the phosphate fertilizer industry.

MATERIALS AND METHODS

Samples Collection

The samples of airborne respirable dust, particulate, and gaseous fluoride (static n = 71 and personal n = 36) were collected from various divisions' phosphate fertilizer production industry. Respirable dust, aerosol-containing particulate, and gaseous fluoride were sampled by following NIOSH method 7902, using personal respirable dust sampler SIDEKICK Ex 51 and universal sample pump 224-PCXR4 (make SKC, USA) through filter cassette flitted with 37mm mixed cellulose ester (0.8μm pore size) filter paper and backup impregnated pad at the flow rate of 2.2 $L.min^{-1}$ and 2.0 $L.min^{-1}$ respectively. Initial and final weights of the MCE filter paper were measured using the analytical balance (make Shimadzu). Respirable dust samplers pump for sampling were tagged to the waist of the workers working in the different sections in the industry, whereas the respirable dust samplers filter cassette and cyclone inlet set were kept in the breathing zone of each worker for the entire shift duration of 8 h. For the static monitoring, the samplers were positioned in the breathing zone (periphery of 1.2 to 1.8 m) of the dust-producing source inside the plant with the help of a tripod stand. After sampling, the filter papers and pads were stored and transported to the laboratory for further analysis.

Measurement of Personal and Static Respirable Dust ($PM_{2.5}$) Concentration

All the collected samples were processed for determination of dust content. The concentrations were computed from the gravimetric weight for the respirable dust ($PM_{2.5}$) evaluation, the differences of filter paper weights before and after sampling for an entire shift. The dust concentration was expressed in $mg.m^{-3}$.

$$\text{Respirable dust concentration } (mg.m^{-3}) = \frac{W1 - W2\ (mg) \times 10^3}{\text{Flow rate (L/min)} \times \text{Time (min)}}$$

Where, W1= Initial weight of the filter paper (mg)
W2 = Final weight of the filter paper (mg)

Estimation of Particulate and Gaseous Fluoride

The method 7902 was used to analyze particulate and gaseous fluoride (NIOSH 1994). Fluoride concentration in particulate and gaseous mist samples was analyzed using a pH/ISE benchtop meter (Model: ORION 4 STAR, Thermo Scientific). Before sample analysis, the fluoride Ion Selective Electrode was calibrated using freshly prepared 0.01, 0.1, 1, 10, and 100 ppm working standard concentrations(Orion 940907).

Particulate fluoride: The particulate fluoride collected on mixed cellulose ester (MCE) filter papers was conditioned in the laboratory before analysis. The post-weighed filter papers were transferred to a nickel crucible containing 5 mL of 20% NaOH for fusion. It was kept in the muffle furnace for 1 to 2 minutes at 600-650°C. After this, the residues were cooled and dissolved in a plastic beaker containing 40 mL of deionized water, filtered solution, and added 50 mL TISAB-II solution (Orion 940907) to each aliquot. Adjusted the pH to 5.5 ± 0.2 using 1-2 mL of con. HCl and made up the volume up to 100ml with the deionized water. The equal volume of the sample & TISAB-II solution was maintained, and the ISE electrode was inserted in the beaker containing samples, and measured the fluoride levels in it. The blank and known standards were measured between sample analyses to check quality control. In the spiked samples, recovery observed was 98±2%.

Gaseous fluoride: The absorbed gaseous fluoride in the alkali-impregnated cellulose pads were transferred to 100-mL plastic beakers. Equal aliquots of distilled water and TISAB-II solution were added and kept for 30 min until reduced to pulp. Stirred and mixed continuously on a magnetic stirrer at room temperature for 2-3 min, and then the readings were measured in ppm using an ion-selective electrode.

Data Analysis

The data was analyzed using Statistical Package for Social Sciences (SPSS) software (version 26).

RESULTS AND DISCUSSION

During the monitoring, 107 airborne particulate and gaseous mist samples (static n = 71 and personal n = 36) were collected using personal samplers at three different divisions in the phosphate fertilizer factory. In the static monitoring, the maximum particulate fluoride concentration of 0.074 $mg.m^{-3}$ was obtained in the single super phosphate (SSP) division, whereas in the case of respirable dust, the maximum concentration of 4.72 $mg.m^{-3}$ was observed in granular single super phosphate (GSSP) division, however, the highest mean level of respirable dust was found in SSP division (Table 1). Due to rock phosphate digestion takes place in the SSP division.

In personal monitoring, the highest respirable dust and particulate fluoride concentrations were observed in the SSP division, followed by GSSP and administration (Table 1). In the SSP division, maximum respirable dust and particulate fluoride concentrations were observed as 1.18 and 0.013 $mg.m^{-3}$, respectively. Overall, the respirable dust and particulate fluoride level was higher in static monitoring than in personal monitoring. The reason is that in the static monitoring, the sampler was fixed in one place for 8 hours at workplaces. Whereas in the personal monitoring, the sampler was attached to the workers for 8 hours, these workers moved here and there for their job requirements. Therefore, exposure load may be less compared to static monitoring.

Correlation analysis was performed to test the relationship between fluoride concentrations and respirable dust concentration. A significant positive correlation ($r > 0.36$, $p < 0.01$) was found between the particulate fluoride and respirable dust collected in the industrial area. This indicates that the anthropogenic fluoride is higher in industry compared to the periphery of the industry.

Table 2 shows that varied levels of gaseous fluoride concentration were observed in the different divisions in static and personal monitoring methods. Generally, the maximum gaseous fluoride concentration was observed in the SSP division have to remove at 2.44 ppm in the static and personal monitoring, followed by GSSP and administration

Table 1: Respirable dust and particulate fluoride concentration ($mg.m^{-3}$) at different divisions in the fertilizer industry.

Monitoring type	Static Monitoring n = 71			Personal Monitoring n = 36		
Divisions	SSP n = 30	GSSP n = 30	Administration n = 11	SSP n = 16	GSSP n = 16	Administration n = 4
Respirable dust Concentration ($mg.m^{-3}$) Mean ± SD (Range)	1.37 ± 0.83 (0.27-4.09)	1.03 ± 0.90 (0.13-4.72)	0.20 ± 0.12 (0.02-0.34)	1.18 ± 1.12 (0.24-4.56)	0.85 ± 0.51 (0.9-2.06)	0.30 ± 0.11 (0.17-0.41)
Particulate Fluoride Concentration ($mg.m^{-3}$) Mean ± SD (Range)	0.018 ± 0.015 (0.004-0.074)	0.008 ± 0.008 (0.001-0.043)	0.001 ± 0.0001 (0.001-0.002)	0.013 ± 0.013 (0.002-.046)	0.007 ± 0.003 (0.004-.014)	0.002 ± 0.002 (0.001-0.006)

Table 2: Gaseous fluoride concentration (ppm) at different divisions in the fertilizer industry.

Monitoring type	Static Monitoring n = 71			Personal Monitoring n = 36		
Divisions	SSP n = 30	GSSP n = 30	Administration n = 11	SSP n = 16	GSSP n = 16	Administration n = 4
Gaseous Fluoride (HF) Concentration (ppm) Mean ± SD (Range)	0.808 ± 0.758 (0.069-2.444)	0.403 ± 0.263 (0.069-1.187)	0.026 ± 0.022 (0.007-0.071)	0.356 ± 0.340 (0.084-1.146)	0.258 ± 0.187 (0.077-0.550)	0.011 ± 0.004 (0.009-0.017)

Table 3: Exposure limits for fluoride by different regulatory bodies at the workplace.

Regulatory bodies	Particulate fluoride	Hydrogen Fluoride	Respirable dust
OSHA PEL* 8-hour TWA	2.5 $mg.m^{-3}$	3 ppm	5 $mg.m^{-3}$
NIOSH REL** Up to 10-hour TWA	2.5 $mg.m^{-3}$	3 ppm	5 $m.m^{-3}$
ACGIH TLV*** 8-hour TWA	2.5 $mg.m^{-3}$	0.5 ppm	5 $mg.m^{-3}$
Indian Factories Act 1948-PEL 8-hour TWA	2.5 $mg.m^{-3}$	3 ppm	10 $mg.m^{-3}$

*Occupational Safety and Health Administration Permissible Exposure Limits (OSHA 2017)
**National Institute for Occupational Safety and Health Recommended Exposure Limit (NIOSH 2019)
***American Conference of Governmental Industrial Hygienists Threshold Limit Values (ACGIH 2023)

(Table 2). The maximum gaseous fluoride concentration in static monitoring was observed in the single super phosphate division, which was lower than the OSHA PEL and Indian Factories Act 1948, 8-hour TWA standard value of 3 ppm (Table 3) (OSHA 2017). In the SSP division in the static monitoring, the gaseous fluoride mean concentration was 0.808 ppm, which is higher when compared to the ACGIH TLV- 8 hrs time-weighted average standard of 0.5 ppm (Table 3) (ACGIH 2003). A significant difference ($p<0.001$) in gaseous fluoride levels was found between the divisions in static monitoring, whereas in personal monitoring, no significant differences were observed.

According to Schwarz et al. (2020) study, the maximum particulate and gaseous fluoride levels in the primary aluminum industry (Slovakia) were reported as 10.29 and 6.21 $mg.m^{-3}$, respectively, whereas in our investigations, the maximum particulate fluoride level was 0.074 $mg.m^{-3}$ and 2.44 ppm respectively. In the US, a study by Seixas et al. (2000) reported total fluoride 4.07 $mg.m^{-3}$ and hydrogen fluoride 0.74 $mg.m^{-3}$ personal airborne exposure in the aluminum smelting industry. The current study found a lower level of particulate and gaseous fluoride compared to aluminum smelting industry workers.

Figs. 1 & 2 showed that the percentage of particulate fluoride concentration was present in the respirable dust concentration among the different divisions in the static and personal monitoring. The percentage of particulate fluoride concentration was distributed considerably higher in the SSP division, followed by GSSP and administration. Ramteke et al. (2018) conducted a study on selected commercial phosphate fertilizers and reported higher levels of fluoride content. The phosphate fertilizer industrial environment could be contaminated by fluoride. Dartan et al. (2017) study says that the surrounding area of the phosphate fertilizer

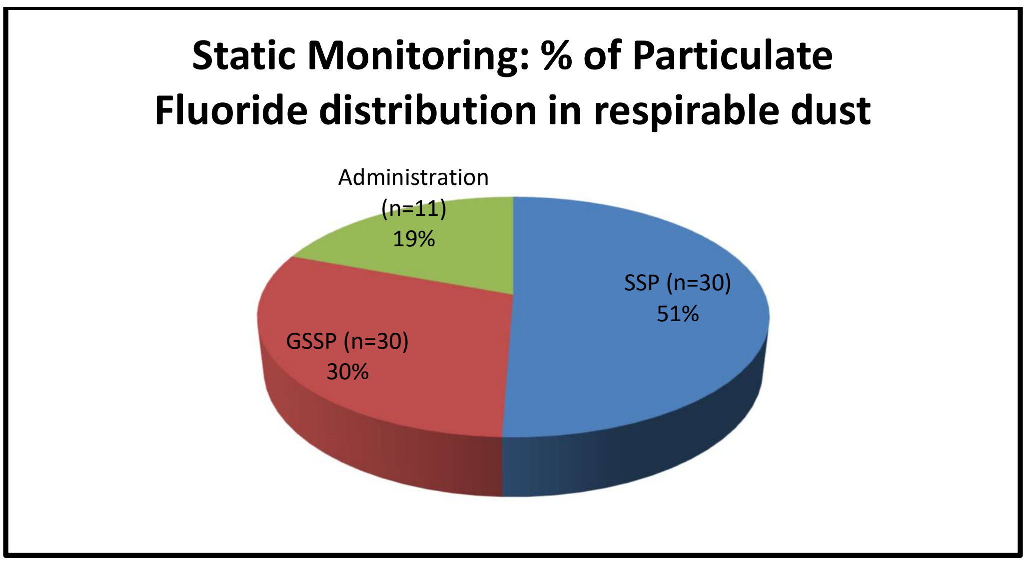

Fig. 1: Percentage distribution of particulate fluoride in the respirable dust (static monitoring).

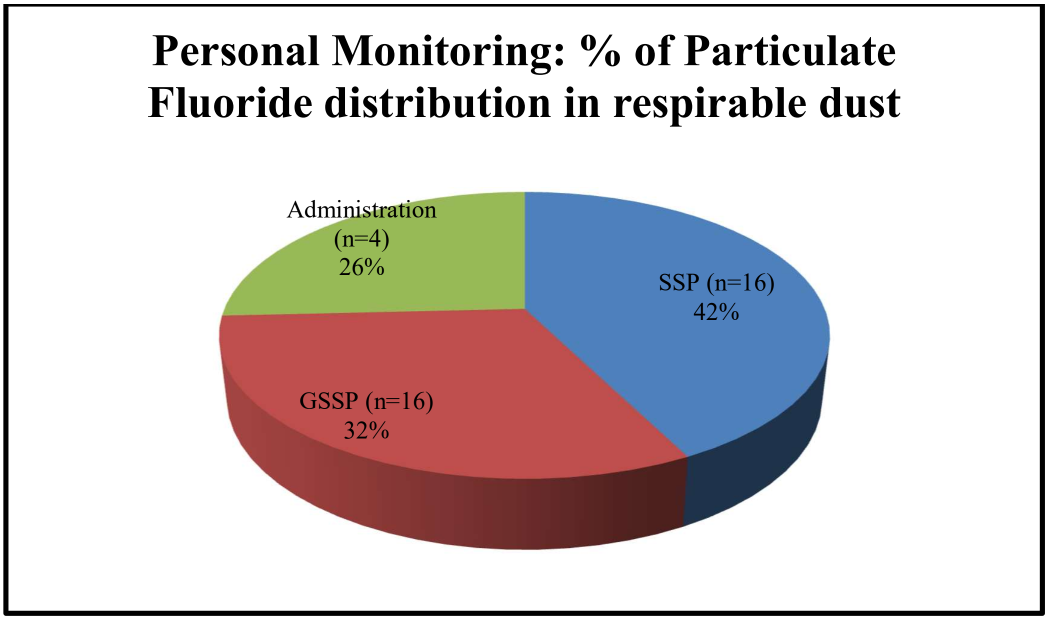

Fig. 2: Percentage distribution of particulate fluoride in the respirable dust (personal monitoring).

industry has serious fluoride contamination. Several previous studies state that chronic fluoride exposure causes adverse effects on bones, skeletal fluorosis, teeth; dental fluorosis, and organs (kidney, liver, thyroid gland, and gastrointestinal); non-skeletal fluorosis in humans and animals (Ghosh et al. 2013, Bhat et al. 2015). Therefore, the workers working in the industry have to avoid fluoride exposure by using personal protective equipment, and rotating the workers' duties from one division to another by the management may help in less exposure.

CONCLUSION

In the present study, we discussed fluoride exposure in the workplace environment in the different divisions of the phosphate fertilizer industry. The results revealed that the concentration of fluoride decreased with the increase of distances from SSP, followed by GSSP and administration. It indicates that the fluoride exposure was indirectly proportional to the distance of the source. The respiratory dust, particulate, and gaseous fluoride concentrations, significant differences ($p<0.001$) were found between the divisions (SSP, GSSP, and administration) of static monitoring. In contrast, in the case of personal monitoring, no significant differences were observed. The percentage distribution of particulate fluoride in the respirable dust in static (51% SSP, 31% GSSP, and 19% administration) and personal monitoring (42% SSP, 32% GSSP, and 26% administration) was observed, respectively. The particulate and gaseous fluoride concentrations obtained were within permissible exposure limits of OSHA, NIOSH, and the Indian Factories Act 1948. Although, we observed a maximum concentration of gaseous fluoride close to the permissible exposure limit in the SSP division. An awareness program was conducted among employees about the effects of excess fluoride exposure and the importance of PPEs in avoiding fluoride exposure in the workplace. Further studies are required on environmental and biological monitoring in the industry and as well as surrounding areas.

ACKNOWLEDGEMENTS

We are expressing sincere thanks to the management and workers of the phosphate fertilizer industry for their cooperation and support during the study. We are thankful to the Director-In-Charge, ICMR-National Institute of Occupational Health, Ahmedabad, for his support. The help of the ROHC(S) team is gratefully acknowledged by the authors.

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