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Effects of uniformities of deposition of respirable particles on filters on determining their quartz contents by using the direct on-filter X-ray diffraction (DOF XRD) method

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ABSTRACT

In this study, field samplings were conducted in three workplaces of a foundry plant, including the molding, demolding, and bead blasting, respectively. Three respirable aerosol samplers (including a 25-mm aluminum cyclone, nylon cyclone, and IOSH cyclone) were used side-by-side to collect samples from each selected workplace. For each collected sample, the uniformity of the deposition of respirable dusts on the filter was measured and its free silica content was determined by both the DOF XRD method and NIOSH 7500 XRD method (i.e., the reference method). A same trend in measured uniformities can be found in all selected workplaces: 25-mm aluminum cyclone > nylon cyclone > IOSH cyclone. Even for samples collected by the sampler with the highest uniformity (i.e., 25-mm aluminum cyclone), the use of the DOF XRD method would lead to the measured free silica concentrations 1.15–2.89 times in magnitude higher than that of the reference method. A new filter holder should be developed with the minimum uniformity comparable to that of NIOSH 7500 XRD method (=0.78) in the future. The use of conversion factors for correcting quartz concentrations obtained from the DOF XRD method based on the measured uniformities could be suitable for the foundry industry at this stage.

1. Introduction

Assessing human exposures to free crystalline silica (including quartz, tridymite, and cristobalite) requires the use of a sampling train consisting of a respirable aerosol pre-selector for allowing particles of the respirable fraction to pass through, and a filter cassette for the particle collection purpose. For each collected sample its crystalline silica content can be determined by using either the NIOSH 7500 X-ray diffraction (XRD) method [1] or the NIOSH 7602 infrared (IR) spectrophotometer method [2]. In principle, the former is more widely used than the latter in the occupational hygiene field because of its higher precision and accuracy [3]. However, it should be noted that the NIOSH 7500 XRD method requires a series of pretreatment processes for each collected sample, including the high temperature ashing, re-suspension, and depositing the collected particles uniformly on a silver membrane filter prior to the XRD analysis [4]. The above pretreatment processes are known for with several disadvantages, including the time consuming, particle loss, and in particular, the cost problem associated with the use of a silver membrane for XRD analyses [5].

In 1985, Kohyama proposed a direct on-filter XRD (DOF XRD) method to eliminate the above mentioned pretreatment procedures [6]. Here, the filter is directly placed against a zinc or aluminum plate (to serve as a reflecting material during the XRD analysis), and its crystalline free silica contents is determined by directly measuring the center of the filter by using the XRD technique. Indeed, if all collected respirable dusts are uniformly deposited on the filter, the result obtained from the DOF XRD method would be representative to the silica content collected on the filter.

In 2005, the UK Health and Safety Executive (HSE) recommended another method (MDHS 101) for free silica analysis [7]. In their method, the respirable particles are collected on a

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membrane filter, and its crystalline silica content is directly analyzed by using infrared spectroscopy (IR) or XRD without the involvement of any pretreatment process. Because all collected respirable dusts are not uniformly deposited on the filter, this method uses a dust cloud generator to generate free silica cloud in a testing chamber. The respirable dust samples collected in the testing chamber are used to establish the calibration curve for free silica analyses. The reference standards for the quartz dust include NIST-SRM 1878 and Sikorn F600 (HSE A9950). The NIST-SRM 1878 is known with a mass median aerodynamic diameter (MMAD) of 1.59 μm and 90% particles <3.78 μm [8]. The MMAD of Sikorn F600 is 2.6 μm and its 70% particles are <4.3 μm [9]. In principle, if the particle size distribution found in a given workplace is exactly same as the above two reference standards, the effect associated with the uniformity of the deposition of respirable dusts on filter for XRD analyses would be negligible. However, it can be expected that particle size distributions found in different workplaces could be very different since the involved manufacturing processes in different industries could be varied greatly.

In this study, respirable dust samplings were conducted in three workplaces with different manufacturing processes. For each collected sample, the uniformity of the deposition of respirable dusts on the filter was measured, and its free silica concentration was determined first by the DOF XRD method and subsequently by the NIOSH 7500 XRD method. The difference in free silica concentrations determined by the above two analytical methods was examined and explained. Then, the effect of the measured uniformities on determining the free silica content for the DOF XRD method was assessed. Finally, conversion factors for correcting quartz concentrations obtained from the DOF XRD method based on the measured uniformities for the foundry industry was proposed. The results obtained from this study will be helpful for assessing workers' free silica exposures in the field if the DOF XRD method is used for the quantification purpose.

2. Materials and methods

2.1. Selected workplaces and sampling methods

In this study, all field samplings were conducted in a foundry plant. Three workplaces were selected, including the molding, demolding, and bead blasting. For each selected workplace, respirable dust samplings were conducted at four sampling sites, where workers spent most of their time to perform their jobs, for approximately one workshift (~8 h). For each sampling site, three sampling trains containing three different types of respirable dust pre-selector were placed side-by-side at the height of 150 cm to collect respirable dusts of the breathing zone. These three types of respirable dust pre-selector include a 25-mm aluminum cyclone (Cat No. 225-01-01, SKC Inc., Eighty Four, PA, USA), a nylon cyclone (Part No. 456243, MAS, Pittsburge, PA, USA), and a cyclone developed by the Institute of Occupational Safety and Health in Taiwan (denoted as the IOSH cyclone) (Fig. 1). The former two respirable dust pre-selectors are currently used in the occupational hygiene field and their sampling flowrates are specified at 2.5 LPM and 1.7 LPM, respectively. The IOSH cyclone (sampling flowrate = 1.7 LPM) was developed to solve the dust overloading and electrostatic problems associated with the use of the former two dust pre-selectors [10].

2.2. Sample analysis

In this study, the mass concentrations of all collected samples were first determined per NIOSH Method 0600 [11]. Then, their crystalline silica contents (including tridymite, cristobalite, and quartz) were determined by both the DOF XRD method [6] and the NIOSH 7500 XRD method [1]. Since the quartz was the only detectable content, and hence only the quartz concentration was measured for each collected sample. The above result was consistent with the results found in our precious study [12]. The calibration curve of the quartz content, as recommended by Verma and Shaw [13], was made by using the NIST-SRM 1878. The scanning range of the 2θ was from the 15° to 65°. The scanning speed was 1°/min and the step size was 0.01°. The calibration curve covered the range from 25 μg to 1000 μg with an R^2 0.997. By analyzing three samples with the same concentration, the accuracy and precision were found as 7.7% and 3.8%, respectively. The instrument stability of XRD, by analyzing 11 samples on different days, was found as 8%. This study yielded the method of detection limit (MDL) of 0.008 mg for quartz.

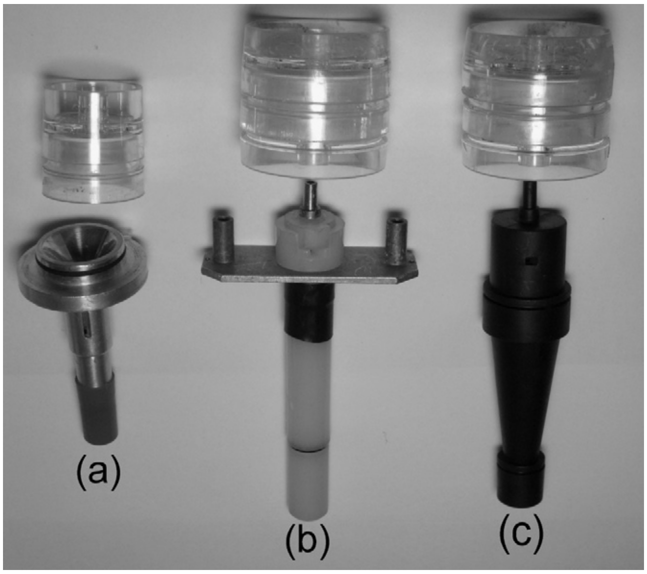

Fig. 1. Three selected sampling trains consist of one of the three types of cyclone, including a (A) 25-mm aluminum cyclone, (B) nylon cyclone, and (C) IOSH cyclone, and followed by a filter cassette.

2.3. Uniformity analysis

In this study, the deposition of respirable particles on a filter was examined using a phase contrast microscope under 400 times of magnification. This study adopted the equal distance as the counting protocol to determine the uniformity of the deposition of respirable particles on a filter. The counting method of equal distance was along each of the radial lines, and each counting field was selected at 0.5 mm intervals. The maximum number of radial lines was four, and maximum counting fields were 200 with minimum intervals of 0.5 mm. After the image processing, the "coverage" (C; i.e., the fraction of total viewing area occupied by the particles) could be used to indicate the ratio of the field area occupied by the particles, and was defined as:

$$C = \left(\frac{P}{F}\right) \times 100\%$$

where P: pixels of all particles in a counting field and F: total pixels in a counting field.

With the coverage of each counting field calculated, the coefficient of variation (CV) could be used to indicate the variation of coverage among counting fields. The CV was given as follows:

$$CV = \left(\frac{\sigma}{\mu}\right)$$

where σ is standard deviation of coverage of the particle area and μ is the mean coverage of the particle area. The uniformity (U) was defined as the following equation:

$$U = e^{-CV}$$

The exponential of the negative CV value was a transformation to easily understand the uniformity of the filter deposits. The U value has limited the variation in a small range from 0 to 1. The image processing method is a tedious and time consuming procedure since there are still many occasions in need of human decision. Therefore, the standard error (SE) of CV was evaluated by the bootstrap re-sampling method [14–16]. The Bootstrap method requires renumbering all the selected counting fields and randomly re-sampling the counting fields. For every re-sampling the CV of the coverage (of the re-sampled counting fields) is recalculated. The bootstrap method can then be expressed as follows:

$$\begin{array}{l} n_1^1 n_2^1 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots n_k^2 \rightarrow CV^1 \\ n_1^2 n_2^2 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots n_k^2 \rightarrow CV^2 \\ . \\ . \\ {n_1}^b {n_2}^b \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots {n_k}^b \rightarrow CV^b \end{array}$$

$$SE(\widehat{CV}) = \sqrt{\frac{1}{B-1}\sum_{b=1}^{B}(CV^b - \overline{CV})^2}$$

where $SE(\widehat{CV})$ is standard error of the CV; $\overline{CV}$ is mean of CV; n_k^b is the calculated particles coverage of the bth image obtained from the kth sampling. In the present study both b and k were specified as 100.

Following the same technique, the uniformity could be evaluated for its standard error using the bootstrap method.

$$SE(\hat{U}) = \sqrt{\frac{1}{B-1}\sum_{b=1}^{B}(U^b - \overline{U})^2}$$

where $SE(\hat{U})$: standard error of U, $\overline{U}$: mean of U.

Detailed calculating procedures can also be seen in our previous publication [17].

3. Results and discussion

3.1. Respirable dust concentrations of the three selected workplaces

Fig. 2 shows the respirable dust concentrations found in the three selected workplaces. By taking the 25-mm aluminum cyclone as an example, it can be seen that the magnitudes of the concentrations (mean ± SD) presented in sequence for the three selected workplaces were: demolding (8.35 ± 1.19 mg/m^3) > bead blasting (1.18 ± 0.99 mg/m^3) > molding (0.67 ± 0.35 mg/m^3). The same trend can also be seen in results obtained from the nylon cyclone and IOSH cyclone (Fig. 2). The highest concentration found in the demolding area could be because hand tools were used directly to crack casting molds and dusts were generated via mechanical forces. Although the bead blasting process also involved in the use of mechanical forces (i.e., to remove debris from the casting object), the installation of an enclosure device would effectively lessen the emissions of dusts to the workplace atmosphere. The lowest concentration found in the molding area might be because the wet process was used in the molding process.

We further compare the respirable dust concentrations collected by the three selected sampling trains from each selected workplace. By taking the molding area as an example (Fig. 2), no significant difference (Kruskall Wallis test, p-value = 0.59) can be found among the concentrations collected by the 25-mm aluminum cyclone (0.67 ± 0.35 mg/m^3), nylon cyclone (0.74 ± 0.37 mg/m^3), and IOSH cyclone (0.71 ± 0.19 mg/m^3). No significant difference can also be seen in the results obtained from both the demolding area and bead blasting area (Kruskall Wallis test, p-value = 0.43 and 0.42, respectively) (Fig. 2). The above results suggest that the three selected cyclones had a very similar performance as a pre-selector for collecting respirable dusts. Here, it should be noted that the IOSH cyclone has been addressed to have the advantage in overcoming particle electrostatic and overloading problems during the sampling process [10]. In the present study, the similar performance found in the three selected cyclones might because respirable dust concentrations were not particularly high in the three selected workplace atmospheres.

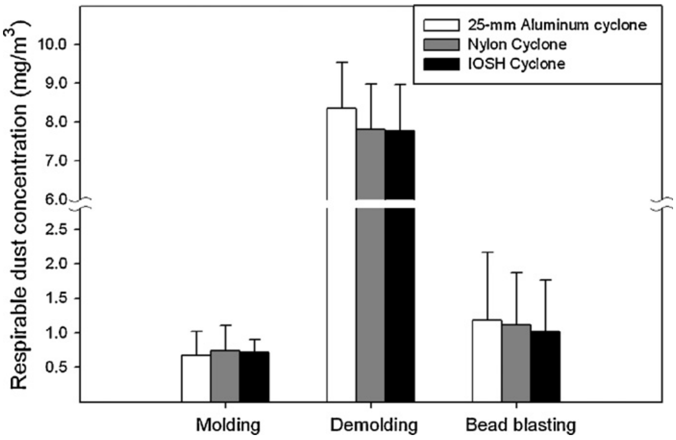

Fig. 2. The respirable dust concentrations (n = 4) obtained from the three selected workplaces by using three different types of cyclone.

3.2. Respirable quartz concentrations of the three selected workplaces

Fig. 3a shows the respirable quartz concentrations (mean ± SD) of the three selected workplaces by using the NIOSH 7500 XRD method. Again, by taking the 25-mm aluminum cyclone as an example we found that: demolding (3.45 ± 0.38 mg/m^3) > bead blasting (0.50 ± 0.20 mg/m^3) > molding (0.27 ± 0.11 mg/m^3). The same trend can also be seen in results obtained from the nylon cyclone and IOSH cyclone (Fig. 3a). Similarly, Fig. 3b shows the respirable quartz concentrations of the three selected workplaces by using the DOF XRD method. Not so surprisingly it can be seen that the concentrations in magnitude presented in sequence for the three selected workplaces (by taking the 25-mm aluminum cyclone as an example) were: demolding (5.16 ± 1.24 mg/m^3) > bead blasting (0.57 ± 0.12 mg/m^3) > molding (0.41 ± 0.09 mg/m^3). The same trend can also be found in concentrations obtained from the nylon cyclone and IOSH cyclone (Fig. 3b). Here, it should be noted that the above trend is the same as that found in respirable dust concentrations (Fig. 2). Obviously, this is because the emitted respirable dusts contain the same quartz content for samples collected from the three selected workplaces (i.e., the same silica sand was involved in the three selected workplaces). On the other hand, very different quartz concentrations were found for samples collected by the same sampling train but analyzed by different methods (i.e., the NIOSH 7500 method and DOF XRD method, respectively) (Fig. 3a and b). The above results warrant the needs for further discussion (see the following section).

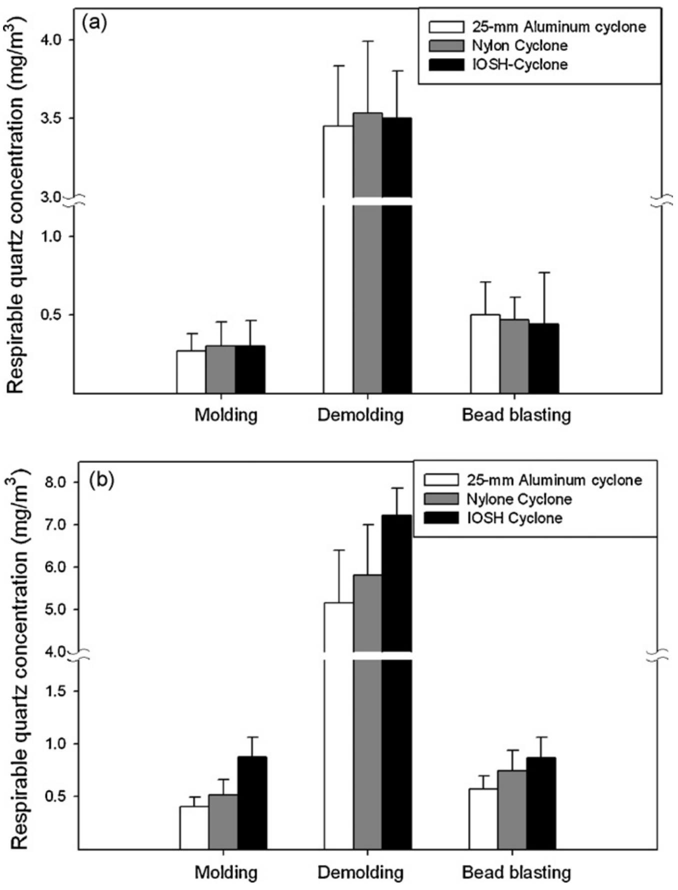

Fig. 3. The respirable quartz concentrations ($n = 4$) analyzed per (a) NIOSH 7500 XRD method, and (b) DOF XRD method for samples collected from the three selected workplaces by using three different types of cyclone.

3.3. Comparisons of respirable quartz concentrations obtained from the NIOSH 7500 and DOF XRDs

In this part of the study, we first compare the respirable quartz concentrations for samples collected from the same workplace using the three different types of sampling trains by using the NIOSH 7500 XRD method. By taking the molding area an example (Fig. 3a), no significant differences (Kruskall Wallis test, p-value = 0.84) can be found among the concentrations collected by using the 25-mm aluminum cyclone ($0.27 \pm 0.11\,\mathrm{mg/m^3}$), nylon cyclone ($0.30 \pm 0.15\,\mathrm{mg/m^3}$), and IOSH cyclone ($0.30 \pm 0.16\,\mathrm{mg/m^3}$). The same trend can also be seen in the results obtained from both the demolding area and bead blasting area (Kruskall Wallis test, p-value = 0.96 and 0.88, respectively) (Fig. 3a). The above results further confirm that the three selected cyclones had a similar performance as a pre-selector for collecting respirable dusts.

As we compare the respirable quartz concentrations for samples collected from the same workplace using the three different types of sampling train but analyzed by the DOF XRD method, a totally different trend is found as in comparison with that analyzed by the NIOSH 7500 XRD method. By taking the molding area an example (Fig. 3b), although no significant differences (Kruskall Wallis test, p-value = 0.06) can be found among the concentrations collected by using the 25-mm aluminum cyclone ($0.48 \pm 0.09\,\mathrm{mg/m^3}$), nylon cyclone ($0.52 \pm 0.14\,\mathrm{mg/m^3}$), and IOSH cyclone ($0.87 \pm 0.19\,\mathrm{mg/m^3}$). It should be noted that the concentrations obtained from the DOF XRD method are consistently greater the corresponding concentrations of the NIOSH 7500 XRD method (Fig. 3a). Similar results can also be found for those samples collected from the demolding area and bead blasting area (Kruskall Wallis test, p-value = 0.15 and 0.18, respectively) (Fig. 3b).

It is known that a series of pretreatment procedures are needed in the NIOSH 7500 XRD method before the XRD analysis can be used for quantification. But these procedures have the advantage to ensure that respirable dusts to be uniformly deposited on the silver membrane filter. On the other hand, because no pretreatment procedure is required in the DOF XRD method and hence might result in non-uniformity of the deposition of respirable dusts on the filter due to their intrinsic particle inertial effects [18]. Particularly, it should be noted these particles tend to deposit on the center of the filter. It is known that the use of the XRD analysis is not required to measure the entire filter area. Instead, only the central spot of the filter (measured area = 13.44 mm × 10 mm) is measured for the quantification of the free silica content. Therefore, it is not so surprising to see that the concentrations obtained from the DOF XRD method were higher than those of the NIOSH 7500 XRD method for samples collected from the same workplace atmosphere.

3.4. Effects of uniformity of the deposition of respirable particles on filters on quantifying quartz concentrations by using the DOF XRD method

As described in the previous section, we found that the use of the DOF XRD method will result in bias in quantifying quartz content collected on filters. Even for samples collected from the same workplace, the use of different types of cyclones as a pre-selector would also result in different respirable quartz concentrations. Therefore, it raises the question regarding the effects of uniformities of the deposition of respirable particles on filters on quartz quantification if the DOF XRD method was used for sample analyses.

Table 1 shows the ratios of the respirable quartz concentrations analyzed by the DOF XRD method to the corresponding concentrations analyzed by the NIOSH 7500 XRD method (to serve as the reference). For the comparison purpose, Table 1 also records the uniformities of the depositions of respirable dust on filters for samples collected from the three selected workplaces by using three different types of sampling train. We found that the resultant uniformities presented in sequence for samples collected by the three different types of sampling train from the three selected workplaces are: 25-mm aluminum cyclone (0.49 ± 0.01–0.57 ± 0.05) > nylon cyclone (0.39 ± 0.09–0.41 ± 0.12) > IOSH cyclone (0.34 ± 0.03–0.37 ± 0.03). The highest uniformities were found in the 25-mm aluminum cyclone might because a bell-shape outlet is used in the cyclone before connecting to the subsequent filter cassette (Fig. 1a). This gradual expansion facility leads to respirable particles become less separated from the air stream and hence results in higher uniformities of particles deposited on filters. On the other hand, both the nylon cyclone and IOSH cyclone do not include a bell-shape outlet. Instead, both directly place the filter cassette on top of the cyclone (Fig. 1b and c). This abrupt expansion scenario results in particles become much easier to be separated from the air steam and then directly impact on the center of the filter due to their inertial effects. Therefore, it is not so surprising to see that the less uniformities were obtained from these two types of cyclone than that of the 25-mm aluminum cyclone. Although the uniformities obtained from the nylon cyclone were still slightly higher than that of the IOSH cyclone, no significant difference was found in the present study.

Table 1 also shows that the ratios of the respirable quartz concentrations obtained from the DOF XRD method to the corresponding concentrations obtained from the NIOSH 7500 XRD method for samples collected by the three types of sampling train from the three selected workplaces. The resultant ratios presented in sequence are: IOSH cyclone (2.07 ± 0.15–2.92 ± 0.33) > nylon

Table 1
The ratios of respirable quartz concentrations obtained from the DOF XRD to the corresponding concentrations obtained from the NIOSH 7500 XRD method, and the uniformities of respirable dusts deposited on filters for samples collected from the three selected workplaces by using the three different types of cyclone ($n = 4$).

Workplace	25-mm aluminum cyclone		Nylon cyclone		IOSH cyclone	
	Ratio	Uniformity	Ratio	Uniformity	Ratio	Uniformity
Molding	1.31 ± 0.20	0.49 ± 0.01	1.71 ± 0.15	0.39 ± 0.09	2.89 ± 0.29	0.34 ± 0.12
Demolding	1.23 ± 0.23	0.57 ± 0.05	1.36 ± 0.27	0.41 ± 0.12	2.07 ± 0.15	0.37 ± 0.03
Bead blasting	1.15 ± 0.18	0.54 ± 0.14	1.60 ± 0.11	0.40 ± 0.08	1.98 ± 0.28	0.34 ± 0.03

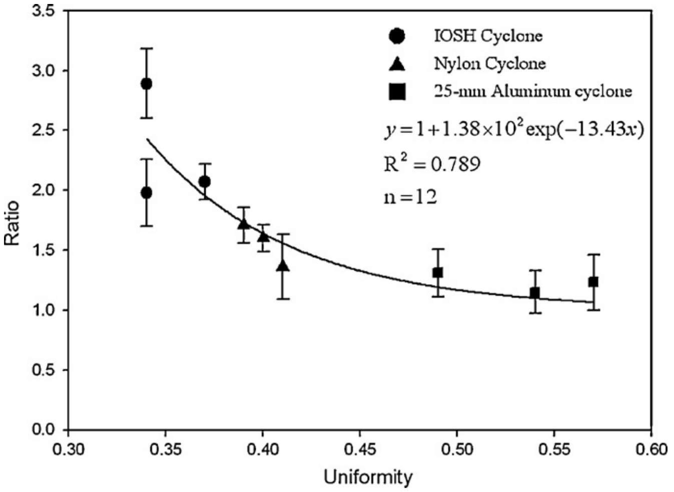

Fig. 4. The relation between the ratios (y) of respirable quartz concentrations obtained from the DOF XRD to that obtained from NIOSH 7500 XRD method and the uniformities (x) of respirable dusts deposited on filter for samples collected by the three types of sampling train from the three selected workplaces.

cyclone (1.36 ± 0.27–1.71 ± 0.15) > 25-mm aluminum cyclone (1.15 ± 0.18–1.31 ± 0.20). Obviously, cyclones with higher uniformities had lower ratio values. But even for the 25-mm aluminum cyclone (known with the highest uniformities), its respirable quartz concentrations obtained from the DOF XRD method were still 1.15 ± 0.18–2.92 ± 0.33 times in magnitude higher than that of the NIOSH 7500 XRD method. Our results clearly indicate that the use of the DOF XRD method for respirable quartz quantification would result in overestimation of workers' exposures.

In this study, no significant difference was found in the resultant respirable quartz concentrations for samples collected from the same workplace using different types of sampling trains if the NIOSH 7500 XRD method was used for sample analyses. Therefore, the uniformity of the deposition of respirable particles on silver filter was chosen as the reference standard. In the present study, the reference uniformity of 0.78 ± 0.02 was yielded based on the results obtained for all collected samples. Obviously, the above values were much higher than that of 25-mm aluminum cyclone (0.49 ± 0.01–0.57 ± 0.05), nylon cyclone (0.39 ± 0.09–0.41 ± 0.12), and IOSH cyclone (0.34 ± 0.03–0.37 ± 0.03). Therefore, if the DOF XRD method will be used for quantifying free silica contents in the future, the development of a new filter holder with its resultant uniformity comparable to that of the NIOSH 7500 XRD method (=0.78) would be necessary.

Fig. 4 shows the ratios (i.e., y) of respirable quartz concentrations obtained from the DOF XRD method to that from the NIOSH 7500 XRD method and their corresponding uniformities (i.e., x) for samples collected from the three selected workplaces using the three types of sampling train. In principle, as x increases to 100% and the values of y would be very close to 1.0 (i.e., respirable quartz concentrations obtained from the DOF XRD method is equivalent to that from the NIOSH 7500 XRD method). Based on the above principle, the following model was used to describe the relationship between x and y:

$$y = 1 + a\,\exp(-bx)$$

By using the nonlinear regression analysis, this study yielded the values for a and b as 1.38×10^2 and 13.43, respectively ($n = 12$, $R^2 = 0.789$). Before new filter holders are developed, the values of y can be served as conversion factors for correcting quartz concentrations obtained from the DOF XRD method based on the measured uniformities (i.e., x) for the foundry industry.

4. Conclusions

For any given workplace, no significant difference was found among the respirable dust concentrations collected by the three types of sampling train suggesting that all three selected cyclones shared a very similar performance as a pre-selector for collecting the respirable dusts. Similar results can also be found in the corresponding respirable quartz concentrations for samples analyzed by using the NIOSH 7500 XRD method, but not for the DOF XRD method. By using the ratios of the respirable quartz concentrations obtained from the DOF XRD method to the corresponding concentrations obtained from the NIOSH 7500 XRD method as an index, the resultant ratios share the same trend as: IOSH cyclone > nylon cyclone > 25-mm aluminum cyclone for samples collected from any selected workplace. The above trend can be explained by the uniformities of the depositions of respirable dusts on filters for each type of cyclone. Cyclones with higher uniformities were found with lower ratio values. Although the 25-mm aluminum cyclone had the highest uniformities, the use of the DOF XRD method for sample analyses would lead to the concentration of the quartz content 1.15–2.89 times in magnitude higher than that of the NIOSH 7500 XRD method. It is concluded that a new filter holder should be developed with the minimum uniformity comparable to that of NIOSH 7500 XRD method (=0.78) in the future. At this stage, the use conversion factors for correcting quartz concentrations obtained from the DOF XRD method based on the measured uniformities might provide useful approach for the foundry industry.

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