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# Trend of utilizing silica fume in concrete and Japanese industrial standard (JIS A 6207)

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## ABSTRACT

Silica fume was well known to contribute strength, durability and workability to concrete in the late 1980s in Japan. As practical performance of silica fume concrete and import volume were increased, Japanese industrial standard (JIS) for silica fume (JIS A 6207) was established in 2000 after studied “Japan society of civil engineers (JSCE) Recommendation for practice of concrete with silica fume” and “Architectural institute of Japan (AIJ) Guideline for mix proportion and construction of concrete using silica fume” in addition to EN13263 Silica fume for concrete-definitions, requirements and conformity control. JIS A 6207 was revised in May 2011, which major revision is water/binder ratio (W/B) in activity index test.

In addition, findings of silica fume quality in Japan and abroad (including content of trace elements and water-soluble trace elements), findings of overseas standards and study results of chemical analysis by x-ray fluorescence are listed.

**Keywords.** Silica fume, Japanese Industrial Standard, Activity index, XRF, Toxics elements

## INTRODUCTION

Silica fume is a by-product collected off-gas from smelting furnace producing elemental silicon, ferrosilicon or alloys containing silicon. It is ultrafine spherical powder which average particle size is 0.1 micron and the specific surface area is about $20m^2/g$. The dominant component is silicon dioxide ($SiO_2$). It is amorphous silica particle soluble in alkaline solution and pozzolanic reaction in concrete is expected.

Producing metallic silicon and ferrosilicon require significant electricity to operate smelting furnace, thus the most production is located at abundance of power such as Scandinavia, Canada and USA. Therefore, silica fume is put into practical use extensively for admixture of concrete or replacement in those areas in order to utilize such by-product.

Figure 1. shows capacity of silica fume production and actual number in 2006 country-by-country. It tells that silica fume production capacity in China increased in addition to Scandinavia and USA in that year. At that day, facility of collecting silica fume was not installed enough to cover production, hence more volume and supplibility are expected today.

Silica fume has been built awareness of effectiveness for improving durability, workability and strength in concrete since in the late 1980s increasing performance record to use and development of supply chain to import. Japanese Industrial Standard (JIS) of silica fume was established in 2000 because of the situation and it has been revised twice.

This paper reports on development of JIS revision and subjects which are conducted a study for next revision.

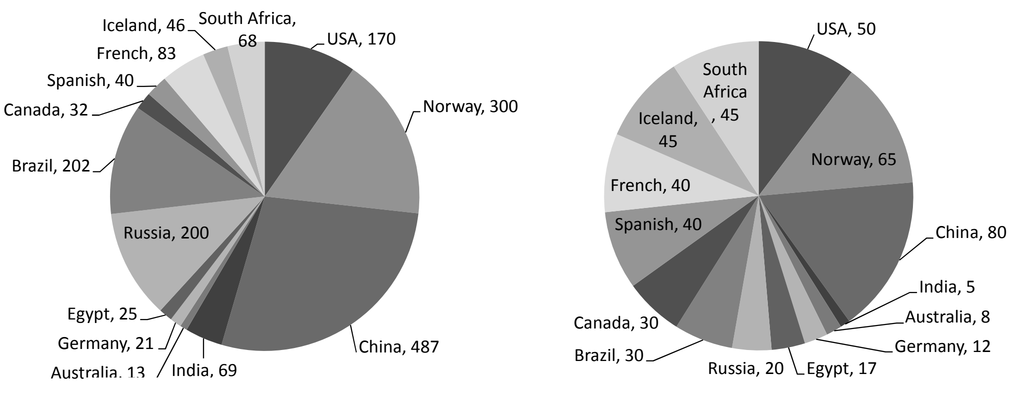


**Figure1. Silica fume production capacity and production volume country-by-country in 2006**

## JIS A 6207 : SILICA FUME FOR USE IN CONCRETE

### Establishment and revision

The first specification for silica fume in JIS was established in 2000.And the first revision was made in 2006. It was realized that producing countries of silica fume sold in Japanese market in 2006 were different from ones in 2000. Thus evaluation of available silica fume and the silica fume concrete with several testing were conducted. According to the test results, the original specification and evaluation method are appropriate to judge quality of silica fume and that concrete. Therefore it was unnecessary to consider substantial revision. However some silica fume samples were not able to pass activity index test even though compressive strength was high enough. Thereby an effective evaluation method regarding to determination of activity index was remained an issue. In addition, X-ray Fluorescent Spectrometer (XRF) analysis was discussed to analyze silica fume in order to expedite analysis work. However, just some assay data were placed in commentary because of few data for the discussion at that time.

The last revision in 2011, 18 samples including out of JIS specification were collected in the global market. Those samples were investigated in quality, in addition, silica fume concrete used 11 samples out of the 18 samples were checked in several testing considering subjects discussed in 2006 revision. In consequence, standard of ignition loss was revised, in addition, the procedure of activity index test and ignition loss test were modified. On the other hand, examining the application of XRF analysis was done with collecting foreign intelligence and checking several analytical methods best suited to silica fume. However, it did not reach standardization. Besides result of study for trace components in silica fume was noted in commentary.

Investigation of trend of standardization in foreign countries and ISO movement was done before issued in 2011 in addition to checking for conflicts between local regulations and new version and confirmed any extinct standards were not adduced as well as revision work in 2006.

## Revision in 2011

## Standard in foreign countries

Various standards for silica fume are available in country to country but ISO standard is not issued yet. Most of the standards are made for silica fume in concrete as admixture. The latest standard in the world is EN13263 in 2005. JIS has the largest number of items to check compared to other standards. Commonly-observed feature among standards is that $SiO_2$, $SO_3$, $Cl^-$, Ignition loss and specific surface (BET) should be specified as important element. Most of standards set down $SiO_2$ > 85%. However only EN standard set 2 classes such as class 1 ($SiO_2$ >85%) and class 2 ($SiO_2$: 80 - 85%). The reason should be beneficial use for low $SiO_2$ to avoid disposal. Some foreign markets refer to EN or ASTM for instance in Middle East. Chinese standard is kind of recommendation but another standard specifying $SiO_2$ > 90% for marine and dam construction is available in China. One of item unintegrated among standards is activity index. JIS set 7 days and 28 days, but ASTM, EN, China and Korea specify one of those.

## Silica fume quality

Silica fume quality investigation in the revision used 18 samples including out of specification to JIS in addition to undensified fume and densified fume. See Table 1. and Table 2. on quality testing. Almost half of the samples have higher chloride against JIS specification ($Cl^-$ < 0.10%) and the highest one is 1.228%. Some samples do not pass activity index test at 28 days more than 7 days. Few samples do not pass ignition loss and all samples have pleasant result on moisture specification $H_2O$ < 1.0%. Most of samples are satisfied with specific surface but one sample is out of specification less than 10 $m^2/g$ that is made from a different production process. The test confirms sulfur trioxide within specification in the all samples. From the results of series of studies, various silica fumes are available in domestic and world market. And some are not satisfied with JIS standard is confirmed.

**Table 1. Test results of 18 samples (Chemical analysis)**

| Silica fume No. | Silica fume Kind | $SiO_2$ (%) | MgO (%) | $SO_3$ (%) | F-CaO (%) | F-Silicon (%) | $Cl^-$ (%) | Ignition loss (%) | Moisture (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | powdered | 83.3[a)] | 3.67 | 0.57 | <0.01 | 0.12 | 0.198[a)] | 3.98 | 0.93 |
| 2 | granulated | 82.7[a)] | 2.39 | 0.70 | <0.01 | 0.04 | 1.228[a)] | 5.49[a)] | 0.88 |
| 3 | powdered | 80.9[a)] | 5.58[a)] | 0.37 | <0.01 | 0.06 | 0.203[a)] | 4.95 | 0.77 |
| 4 | granulated | 90.3 | 2.10 | 0.11 | <0.01 | 0.04 | 0.072 | 2.62 | 0.66 |
| 5 | powdered | 88.7 | 2.26 | 0.13 | <0.01 | 0.02 | 0.057 | 2.71 | 0.73 |
| 6 | powdered | 97.3 | 0.26 | 0.22 | <0.01 | 0.04 | 0.017 | 1.06 | 0.30 |
| 7 | powdered | 96.0 | 0.21 | 0.15 | <0.01 | 0.10 | 0.21[a)] | 2.09 | 0.23 |
| 8 | powdered | 95.2 | 0.62 | 0.13 | <0.01 | 0.05 | 0.028 | 1.48 | 0.26 |
| 9 | powdered | 94.5 | 0.89 | 0.49 | <0.01 | 0.03 | 0.391[a)] | 1.65 | 0.27 |
| 10 | powdered | 92.0 | 0.54 | 0.23 | <0.01 | 0.04 | 0.071 | 3.23 | 0.35 |
| 11 | powdered | 94.0 | 0.19 | 0.38 | <0.01 | 0.15 | 0.016 | 5.07[a)] | 0.38 |
| 12 | powdered | 92.9 | 0.03 | 0.03 | <0.01 | 0.03 | 0.018 | 0.84 | 0.18 |
| 13 | powdered | 91.4 | 1.40 | 0.39 | <0.01 | 0.12 | 0.194[a)] | 3.21 | 0.50 |
| 14 | powdered | 84.7[a)] | 2.20 | 0.49 | <0.01 | 0.09 | 0.477[ca] | 5.12[a)] | 0.79 |
| 15 | powdered | 87.5 | 1.98 | 0.88 | <0.01 | 0.11 | 0.273[a)] | 5.45[a)] | 0.61 |
| 16 | granulated | 96.9 | 0.12 | 0.46 | <0.01 | 0.09 | 0.018 | 1.53 | 0.18 |
| 17 | granulated | 88.9 | 2.27 | 0.63 | <0.01 | 0.11 | 0.184[a)] | 2.95 | 0.61 |
| 18 | granulated | 87.8 | 1.87 | 1.10 | <0.01 | 0.39 | 0.068 | 3.79 | 0.86 |
| Max. value | | 97.3 | 5.58 | 1.10 | 0.0 | 0.39 | 1.228 | 5.45 | 0.93 |
| Min. value | | 80.9 | 0.03 | 0.03 | 0.0 | 0.02 | 0.016 | 0.84 | 0.18 |
| Ave. value | | 90.3 | 1.59 | 0.414 | 0.0 | 0.37 | 1.212 | 3.12 | 0.527 |
| Standard deviation | | 1.19 | 0.340 | 0.287 | 0.0 | 0.091 | 0.207 | 1.50 | 0.187 |
| Quality standard [b)] | | 85≤ | ≤5.0 | ≤3.0 | (≤1.0) | (≤0.4) | (≤0.10) | ≤5.0 | ≤3.0 |

[a)] Out of specification [b)] Quality standard by JIS A 6207 : 2006

**Table 2. Test results of 18 samples (physical properties)**

| Silica fume No. | Kind | Specific Surfaces Area ($m^2/g$) | Density ($g/cm^3$) | Activity index (%) 7 days | 28 days |
|---|---|---|---|---|---|
| 1 | powdered | 13.6 [a)] | 2.20 | 104 | 109 |
| 2 | granulated | 17.1 | 2.20 | 98 | 97 [a)] |
| 3 | powdered | 19.2 | 2.22 | 105 | 106 |
| 4 | granulated | 16.8 | 2.13 | 102 | 104 [a)] |
| 5 | powdered | 18.2 | 2.18 | 108 | 110 |
| 6 | powdered | 19.4 | 2.14 | 104 | 107 |
| 7 | powdered | 20.3 | 2.09 | 106 | 105 |
| 8 | powdered | 15.5 | 2.15 | 96 | 104 [a)] |
| 9 | powdered | 14.2 [a)] | 2.14 | 92 [a)] | 94 [a)] |
| 10 | powdered | 17.2 | 2.15 | 88 [a)] | 92 [a)] |
| 11 | powdered | 30.2 | 2.10 | 105 | 108 |
| 12 | powdered | 8.8 [a)] | 2.17 | 97 | 101 [a)] |
| 13 | powdered | 23.8 | 2.13 | 107 | 106 |
| 14 | powdered | 17.1 | 2.20 | 101 | 100 [a)] |
| 15 | powdered | 23.6 | 2.21 | 104 | 103 [a)] |
| 16 | granulated | 18.4 | 2.09 | 103 | 108 |
| 17 | granulated | 17.3 | 2.22 | 100 | 106 |
| 18 | granulated | 17.1 | 2.20 | 82 [a)] | 80 [a)] |
| Max. value | | 30.2 | 2.22 | 108 | 110 |
| Min. value | | 8.8 | 2.09 | 82 | 80 |
| Ave. value | | 18.2 | 2.16 | 100 | 102 |
| Standard deviation | | 4.55 | 0.044 | 6.95 | 7.47 |
| Quality standard [b)] | | 15≤ | - | 95≤ | 105≤ |

[a)] Out of specification [b)] Quality standard by JIS A 6207 : 2006

## Revision of activity index test

The new standard specify water-binder ratio (W/B) is 0.30 from 0.50 set in the previous standard. See Table 3., modification points in activity index test and Table 4. shows mortar mixture of previous and update standard. For information and reference for the revision, a questionnaire survey was performed to people who work for high strength concrete using silica fume in Japan. According to the information, compressed strength would be 80 - 150MPa and W/B be less than 0.30 when silica fume is applied. Especially if W/B is less than 0.20, it is confirmed that attention to silica fume is increased by means of comments in the survey. As the result of the survey, silica fume is used actively at low W/B for instance 0.20, the authors investigated what water ration should be the best standard to evaluate effectiveness of silica fume in concrete considering results in 2006 revision work. Two activity index tests are used at this time, one is previous JIS method and the other is "Architectural institute of Japan (AIJ) JASS 5 M-701: 2005 (Quality of cement for high strength concrete)".

**Table 3. Comparison table between previous and current additions on activity index test**

| | JIS A 6207 : 2006 | JIS A 6207 : 2011 |
|---|---|---|
| Mixture proportion of test mortar | W/C (W/B) = 0.50 | W/C (W/B) = 0.30 |
| | see Table 4. | |
| Chemical admixture | Not use | Use |
| Mortar flow | - | 260±10mm |
| Air content | - | ≤2.0% |

**Table 4. Comparison table between previous and current additions on mixture proportion of mortar for activity index test**

| | Class of mortar | Cement (g) | Test sample (g) | Standard Sand (g) | Total amount of water and admixture(g) |
|---|---|---|---|---|---|
| JIS A 6207 (2006) | Standard Mortar | 450±2.0 | 0 | 1350±5.0 | 225±1.0 |
| | Test mortar | 405±1.8 | 45±0.2 | | |
| JIS A 6207 (2011) | Standard Mortar | 964±2 | 0 | 1350±5 | 289±1 |
| | Test mortar | 868±1.8 | 96±0.2 | | |

**Table 5. Mixture proportion of test concrete**

| | Water binder ratio | Unit water content (kg/m$^3$) | Unit content of Coarse aggregate (kg/m$^3$) | Slump flow (cm) | Air content (%) |
|---|---|---|---|---|---|
| OP:SF=9:1[a) ] (mass %) | 0.2 | 155 | 865 | 65.0±10.0 | 2.0±1.0 |
| [a)] OP : Ordinary portland cement, SF : Silica fume | | | | | |

Regarding activity index, test samples are picked up 11 samples out of the 18 samples showed in Table 1. and Table 2., and used W/B=0.50 as specified in 2000 and W/B=0.30 at this revision. Figure 3. shows result of activity index in mortar and concrete. Mixture proportion of concrete is showed in Table5.. Figure 4. shows relation between activity index and material age (7 days and 28 days) for mortar and concrete. The result tells an indication that lower W/B provide specimen with better growth of activity index from 7 days to 28 days. The reason could be continuous pozzolanic reaction of silica fume in higher alkali area in concrete due to high unit cement content. For all these reasons, it is concluded that activity index test in mortar with W/B=0.30 is suitable for evaluation of silica fume quality in accordance with current silica fume concrete application in Japan.

On the other hand, here is a possibility for silica fume to apply not only high strength concrete but other area in the future in Japan. It will be necessary to investigate actual performance effectiveness using silica fume and further revision should be essential.

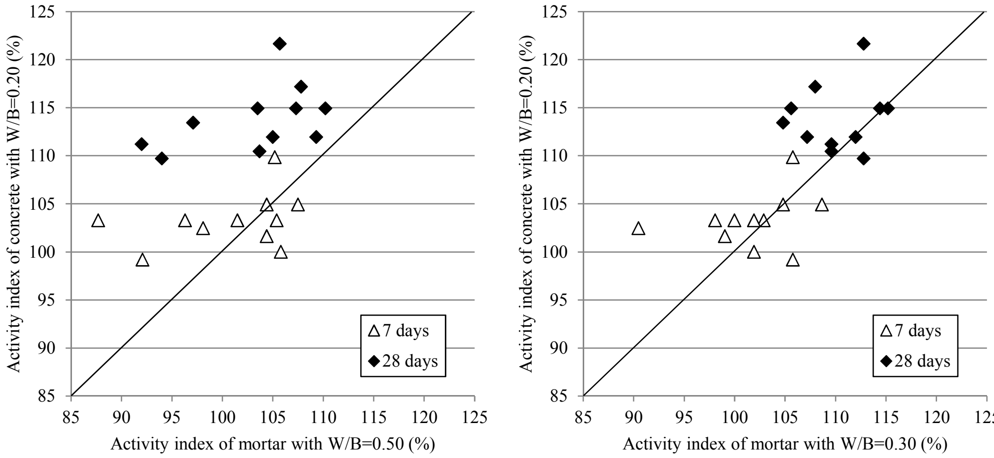


**Figure 3. Relation between activity index of mortar and concrete**

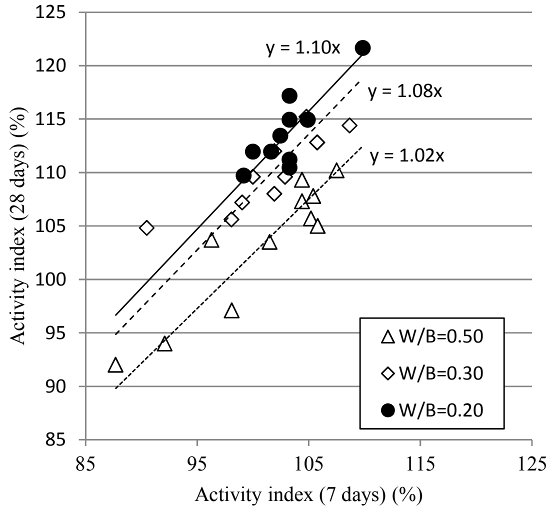


**Figure 4. Relation between activity index of 7 days and 28 days**

## NEXT REVISION

### X-ray Fluorescent Spectrometer analysis (XRF)

In general, XRF analysis is used extensively as analysis for silica fume in foreign countries. However a study of standardization of XRF analysis for silica fume has been shelved in Japan due to lack of information for degree of accuracy to analyzing silica fume with the method.

When powder specimen is analyzed by XRF, pellet formation is normally used for analysis. However, they say that fineness of powder and forming pressure affect precision of analysis compared to glass bead method. Therefore a glass bead of sample preparation was evaluated at 2006 revision work, see Table 6..

The results show that both methods have good correlation even some data have different value. However making glass bead with sample number 7 and 8 was failure. In this case, a

**Table 6. Comparison table between X-ray analysis and chemical analysis**

| No. | X-ray analysis (%) | | | | Chemical analysis (%) | | | |
|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | MgO | $SO_3$ | $Cl^-$ | $SiO_2$ | MgO | $SO_3$ | $Cl^-$ |
| 1 | 96.0 | 0.49 | 0.07 | 0.030 | 96.2 | 0.76 | 0.04 | 0.034 |
| 2 | 96.0 | 0.49 | 0.11 | 0.055 | 96.9 | 0.68 | 0.12 | 0.061 |
| 3 | 94.6 | 0.39 | 0.12 | 0.015 | 95.0 | 0.58 | 0.13 | 0.017 |
| 4 | 94.1 | 0.64 | 0.19 | 0.117 | 94.6 | 0.70 | 0.25 | 0.127 |
| 5 | 84.9 | 1.34 | 1.84 | 0.027 | 85.0 | 1.63 | 1.95 | 0.028 |
| 6 | 88.8 | 1.07 | 0.44 | 0.075 | 92.7 | 1.29 | 0.86 | 0.085 |
| 7 | 95.5 | 0.27 | <0.01 | <0.001 | 95.4 | 0.42 | 0.03 | 0.056 |
| 8 | 90.1 | 0.46 | <0.01 | 0.001 | 93.3 | 0.50 | 0.05 | 0.042 |
| 9 | 83.7 | 1.90 | 0.28 | 1.290 | 84.3 | 1.88 | 0.28 | 1.432 |
| 10 | 96.3 | 0.40 | 0.22 | <0.001 | 96.4 | 0.54 | 0.36 | 0.002 |

**Table 7. XRF analysis results of silica fume standard sample (NIST SRM 2696)**

| Analytical company | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | $P_2O_5$ | $Cl^-$ |
|---|---|---|---|---|---|---|---|---|---|---|
| A | 96.77 | 0.19 | 0.03 | 0.44 | 0.20 | 0.09 | 0.150 | 0.650 | 0.100 | 0.186 |
| B | 97.06 | 0.05 | 0.19 | 0.44 | 0.20 | 0.20 | 0.140 | 0.660 | 0.100 | 0.194 |
| C | 97.03 | 0.25 | 0.02 | 0.46 | 0.24 | 0.21 | 0.170 | 0.670 | 0.100 | 0.215 |
| D | 96.39 | 0.19 | 0.11 | 0.43 | 0.20 | 0.19 | 0.140 | 0.640 | 0.100 | 0.185 |
| Standard Reference Material 2696 | 95.61 | 0.208 | 0.055 | 0.426 | 0.235 | 0.160 | 0.129 | 0.652 | 0.086 | 0.172 |

pretreatment is required for sample, which is heat treatment for instance over 1000 degree C. The treatment result in analysis precision err because $Cl^-$ and $SO_3$ are volatized by heat. In order for the solution, it is considered for value that sample is pelletized. However several conditions to make suitable pellet was unclear at that time. Furthermore it is necessary for standardization of such analytical method to determine standard sample and standard curve, and evaluate data variation and accuracy are needed. Thus, one of important subjects mentioned above, basis of sample preparation for XRF was evaluated in 2011 revision work. Basic data was collected through the work.

According to the result, it is confirmed that pelletizing method is predictably-effective. Because the silica fume standard sample (NIST SRM 2696) was analyzed at 4 different analytical companies and the results are showed in Table 7..

The following subjects should be necessary for stepping toward the practical use of XRF analysis for silica fume. 1) Preparation of enough analytical samples for making standard curve. 2) Investigating effect of sample powder size for analysis accuracy. 3) Accumulation of data from some analytical companies and institutes.

## Toxics elements (trace components) in silica fume

The Soil Contamination Countermeasures Act came into operation in February 2003 in Japan. Demand of reducing or terminating toxic elements in industrial waste from construction related is increased. Toxic elements in cement for example hexavalent chromium is now discussed in construction business therefore such elements in silica fume should be studied and discussed. The authors investigated toxic elements leaching rate from mortar which specimen contains replaced 10% silica fume against Ordinary Portland cement (OPC). The test is what we call "tank leaching test". The subject toxic elements are following 8 elements such as cadmium (Cd), lead (Pb), hexavalent chromium ($Cr^{6+}$), arsenic (As), mercury (Hg), selenium (Se), fluorine (F) and Boron (B).

Number of silica fume sample is 5 which include out of specification against JIS. Meanwhile toxic elements in the sample and leaching rate were checked before the tank leaching test. OPC is used Japanese one commonly used. Table 8. shows toxic elements in the silica fume samples. The all samples meet Japanese environmental regulation in terms of As. However sample A and D exceed allowable limit of Pb. The reason is the sample A and D are out of specification against JIS and contain a lot of contamination. Table 9. shows toxic elements leaching rate in silica fume. Leaching rate of As in all samples exceed Japanese

**Table 8. Results of content test of five silica fume samples**

| | Silica fume sample | | | | | Determination limit value | Environmental standards value |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | F | | |
| Cd(mg/l) | - | - | - | - | - | 0.001 | ≤0.01 |
| Pb(mg/l) | 600* | 39 | 25 | 160* | 85 | 0.005 | ≤0.01 |
| $Cr^{6+}$(mg/l) | | | | | | 0.005 | ≤0.05 |
| As(mg/l) | 57 | 43 | - | - | - | 0.002 | ≤0.01 |
| Hg(mg/l) | - | - | - | - | - | 0.0005 | ≤0.0005 |
| Se(mg/l) | - | - | - | - | - | 0.002 | ≤0.01 |
| F(mg/l) | 480 | 400 | - | - | - | 0.1 | ≤0.8 |
| B(mg/l) | - | - | - | - | - | 0.01 | ≤1 |

- : Less than a determination limit value, * : Excess of environmental standard value

**Table 9. Results of leaching test of five silica fume samples**

| | Silica fume sample | | | | | Determination limit value | Environmental standards value |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | F | | |
| Cd(mg/l) | - | - | 0.019* | 0.004 | 0.022* | 0.001 | ≤0.01 |
| Pb(mg/l) | - | - | 0.021* | 0.018* | 4.3* | 0.005 | ≤0.01 |
| $Cr^{6+}$(mg/l) | 0.12* | 0.01 | - | - | - | 0.005 | ≤0.05 |
| As(mg/l) | - | - | - | - | - | 0.002 | ≤0.01 |
| Hg(mg/l) | 0.099* | 0.064* | 0.02* | 0.25* | - | 0.0005 | ≤0.0005 |
| Se(mg/l) | 18* | 28* | 2.0* | 20* | 0.5* | 0.002 | ≤0.01 |
| F(mg/l) | 3.5* | 1.8* | 0.49 | 0.61 | 0.87 | 0.1 | ≤0.8 |
| B(mg/l) | 9.5 | 9.5 | 7.4 | 7.8 | 2.9 | 0.01 | ≤1 |

- : Less than a determination limit value, * : Excess of environmental standard value environmental control limit considerably. Few samples exceed the allowable limit of Cd, Pb, $Cr^{6+}$, Se, F and B.

Table 10. shows the example of results of the tank leaching test of mortar specimen. Only sample A is tested with W/B=0.50, 0.30 and 0.15. The other samples are W/B=0.30 only. 10% of OPC is replaced by silica fume for all specimens. According to the test results, little elements are eluted from the specimens. It is confirmed that hardened mortar might not be problem in the matter but it requires further study.

**Table 10. Results of tank leaching test of hardened mortar (Plain and using sample A)**

| | W/C (W/B) =0.50 | | W/C (W/B) =0.30 | | W/C (W/B) =0.15 | | Determination limit value | Environmental standards value |
|---|---|---|---|---|---|---|---|---|
| | Plain | With SF:A | Plain | With SF:A | Plain | With SF:A | | |
| Cd(mg/l) | - | - | - | - | - | - | 0.001 | ≤0.01 |
| Pb(mg/l) | - | - | - | - | - | - | 0.005 | ≤0.01 |
| $Cr^{6+}$(mg/l) | - | - | - | - | 0.006 | 0.005 | 0.005 | ≤0.05 |
| As(mg/l) | - | - | - | - | - | - | 0.002 | ≤0.01 |
| Hg(mg/l) | - | - | - | - | - | - | 0.0005 | ≤0.0005 |
| Se(mg/l) | - | - | - | - | - | - | 0.002 | ≤0.01 |
| F(mg/l) | 0.1 | - | - | 0.1 | 0.1 | 0.1 | 0.1 | ≤0.8 |
| B(mg/l) | - | - | - | - | - | - | 0.01 | ≤1 |

SF: Silica fume, -: Less than a determination limit value

## CONCLUSION

It has been over a decade since JIS for silica fume was established and revised twice. During this period, high strength concrete technology is developed rapidly and silica fume is recognized a crucial material for the application in Japan. It is expected in the coming years in Japan that silica fume will be applied to not only high strength concrete but durable one as seen in many foreign.

## REFERENCE

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