Factors Accelerating Dioxin Emission from Iron Ore Sintering Machines

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(Received on September 19, 2008; accepted on December 5, 2008)

Causal factors for dioxin (D) emission from iron ore sintering machines have been investigated by means of pot tests designed with the two-level seven-factor orthogonal array (La(2)^7) and multiple regression analysis on commercial plant data.

EP dust, BF dust and purchased scale were major D-accelerators; quick lime was a D-suppressor. D emission was not a sum but a product event of those factors, and their coefficients were 28, 4, 4 and 0.7 times for 5% EP dust, 5% BF dust, 5% purchased scale and 2% quick lime additions, respectively. As for operational conditions, bed height, coke content, gas velocity and after burning had insignificant effects whereas the increase of hearth layer and the decrease of BTP position increased D emission, which implied the hearth layer and wind boxes could be other synthesis sites.

KEY WORDS: agglomeration; dioxins; iron ore sintering.

1. Introduction

In 1997, Japan, off-gas from iron ore sintering machines contained 0.03 to 2.7 ng-TEQ/Nm^3 dioxins (D), summing to 135 g-TEQ annually, which positioned the sintering plants to the third largest domestic D source.

The Japan Iron and Steel Federation organized a research committee (SDD committee) in 1997 with Japanese universities and integrated steel makers, aiming to clarify the mechanism of D formation during sintering and to propose techniques to suppress its formation in the bed, before they proclaimed the voluntary action program of D decrease in 1999. The four-year activity of the committee gave wide information about D emission, which included plant observations, sintering pot tests to find causal factors, quenching pot test focusing on D formation/transfer behavior in the bed and basic researches to identify D embryo in sintering process.1)

The Japanese government enshrined D decreasing measures into law on July 16, 1999, and forced the existing sinter plants to reduce D concentration below 1.0 ng-TEQ/Nm^3, aiming to cut-down the domestic emission by 90 mass% of approximately 7.5 kg-TEQ annual in 1997. Before the moratorium of the act terminated in 2001, all sintering machines completed actions covering revert material restriction of use and construction of de-D equipment. Since then, they have been keeping the emission under the regulation value. In EU almost the same measures are undertaken according to Boscolo et al.2) review.

Already D issue has calmed down in Japan. But, when the committee was active, available data was limited. A number of data piled up thereafter enough to check their results. In order to advance understanding on the causal factors for D emission of sintering machines, this work pot-tested several factors of ingredients and operational conditions, and made the regression analysis with commercial plant data.

2. Experimental Method

2.1. Pot Test Method

2.1.1. Pot Test Equipment

Figure 1 shows the pot test equipment. The pot had 300 mm in diameter and 600 mm in height, containing 70 kg of sintering raw mixture. The ignition burner supplied 1100°C×90 s LPG flame for start; the blower evacuated exaust gas at constant pressure of 13 kPa. Sampling gas flowed into the D sampling unit through the flange located on the flue duct of 250 mm diameter at 1.5 m downstream from the wind box.

Fig. 1. Pot test equipment.
2.1.2. Gas Sampling Method

JIS K0311 ruled the method of D measurement in wasted gas. Gas sampling unit was assembled under the JIS instruction. Sampling started right after the burner ignited and terminated 5 min after the exhaust gas attained to the maximum temperature.

JIS Z8808 related to the isokinetic suction necessary to ensure the accuracy of data as flying particulates affected the D concentration. In case of sintering under constant suction pressure, exhaust gas velocity started to gain around the burn through point (BTP). Therefore, monitoring the flow rate with the orifice meter before the blower, the pomp power of the sampling unit was adjusted to maintain the isokinetic condition.

2.2. Pot Test Design and Condition

Prior researchers recognized that several revert materials and operational condition affected D emission and that D generated in the bed left for off-gas around BTP. Which implied that BTP position could affect the emission.

Test 1 examined the effects of ingredients, taking EP dust, BF dust, domestic and purchased scales, anthracite and quick lime into experimental conditions, and their contents were varied between two levels of 0 or 5 mass% in a Japanese standard blend of ores: 22 mass% Robe river, 22 mass% Newman, 22 mass% Carajas, 22 mass% CVRD SSF and 12 mass% limestone. Quick lime of 2% was expected to kill an active form of chlorine to CaCl₂.

Test 2 focused on operational conditions. Reducing them to bed height and three heat wave characteristics: frame propagation speed (FFS), maximum heating temperature (Tmax) and cooling speed (CS), we allowed gas velocity, coke content and on/off of after-burner to control the FFS, Tmax and CS, respectively. Specifically, the suction gas velocity changed 0.30 to 0.45 m/s, the coke content varied 4.2 to 4.5 mass% and after-burner switched on or off with the bed height set at 400 mm or 600 mm. As a reference EP dust also changed 0 to 0.05 mass% of averaged dosage for typical operation. Note that Test 2 adjusted suction pressure so as to maintain the given constant gas velocity throughout.

Runs in each test were allocated on the L₈(2⁷) orthogonal array for 2-levels, 7-factors and 8-runs. Tables 1 and 2 show the allocations with the results of D concentrations observed in off gas for Tests 1 and 2, respectively.

2.3. Regression Analyses on Plant Data

Pot test did not simulate sintering plant operation perfectly. Lack of dust collector possibly made the pot-tested D figures larger than those of plants that usually represented the D concentrations at the position after electric precipitator (EP). No circulation of return fines and EP dust could affect the result. Therefore, it was worthwhile to check the pot test results with plant data.

After classifying data into two groups: those of conventional type and those of waste gas circulating one, consisting of 30 and 27 pieces of data, respectively, we made a multiple regression analysis with use of a commercial software (Visual-stat 4.5J) running in forward stepwise mode to extract significant factors under the criteria of 1.0 and more in F-value. We chose the six ingredients same as those in Pot Test 1 and three operational conditions: bed height, hearth layer consumption and BTP position, as explanatory variables. Note, however, that when a coefficient obtained for a variable had shown a reverse effect than expected, the variable was masked and re-calculated.

3. Results

3.1. Test 1

3.1.1. D Data Observed

The last row of Table 1 shows the observed D data of Test 1 with the ratio to the base condition of no revert use. The D data in Test 1 varied from 0.3 ng-TEQ/Nm³ of base to 220 ng-TEQ/Nm³ of Run 6 with admixing scale purchased, BF dust and EP dust together, 733 times of the base.

3.1.2. Conventional Analysis Based on ‘Sum Model’

The orthogonal experiment implicitly assumes the following data structure that a datum (xᵢ) is expressed as the summation of an average (μ), main effects (αᵢ, jk) and an error (εᵢ).

\[ xᵢ = μ + \sum \alphaᵢ, jk + \epsilonᵢ \quad (i = 1 \pm 7) \text{......(1)} \]

Table 1. Error of D measurement for pot test and commercial plant.

| Run No. | 1-1 | 1-2 | 1-3 | 1-4 | 1-5 | 1-6 | 1-7 | 1-8 |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|
| Scale purchased (mass%) | 0 | 0 | 0 | 0 | 5 | 5 | 5 | 5 |
| Scale domestic (mass%) | 0 | 0 | 5 | 5 | 0 | 0 | 5 | 5 |
| BF dust (mass%) | 0 | 0 | 5 | 5 | 5 | 0 | 0 | 0 |
| EP dust (mass%) | 0 | 0 | 5 | 5 | 0 | 0 | 5 | 5 |
| Anthracite (mass%) | 0 | 1.5 | 0 | 1.5 | 0 | 1.5 | 0 | 1.5 |
| Quick lime (mass%) | 0 | 2 | 2 | 0 | 2 | 0 | 2 | 0 |
| Ds (ng-TEQ/Nm³) | 0.3 | 7.8 | 2.2 | 51 | 3.3 | 220 | 2.6 | 43 |
| Ratio to base (±1) | 1 | 24 | 88 | 170 | 11 | 733 | 9 | 143 |

Table 2. Test 1: Experimental design and allocation of factors concerning ingredients with D concentrations in off gas.

| Run No. | 2-1 | 2-2 | 2-3 | 2-4 | 2-5 | 2-6 | 2-7 | 2-8 |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|
| EP dust (mass%) | 0 | 0 | 0 | 0 | 0.05 | 0.05 | 0.05 | 0.05 |
| Coke (mass%) | 4.5 | 4.5 | 4.2 | 4.2 | 4.5 | 4.5 | 4.2 | 4.2 |
| Bed height (mm) | 600 | 400 | 600 | 400 | 600 | 400 | 400 | 400 |
| Gas velocity (m/sec) | 0.30 | 0.45 | 0.45 | 0.30 | 0.30 | 0.45 | 0.45 | 0.30 |
| After-burner (º) | Off | On | On | Off | On | Off | Off | On |
| Ds (ng-TEQ/Nm³) | 0.37 | 0.11 | 0.17 | 0.068 | 0.082 | 0.073 | 0.8 | 0.13 |
\[ \alpha_{i0} + \alpha_{i1} = 0 \] ..........................(2)

\[ E(\varepsilon_k) = 0 \] ..........................(3)

where \( X_k \): observed data for Run \( k \) \((k = 1 - 8)\)
\( \mu \): the average for all Runs
\( \alpha_{ij} \): the main effect of factor \( i \) and level \( j \) \((i = 1 - 7, j = 0 \text{ or } 1)\)
\( \varepsilon_k \): error accompanied with Run \( k \)
\( E(\varepsilon_k) \): expectation for error

The set of data was firstly solved with a conventional method based on the sum model to obtain the main effect of each factor numerically (Fig. 2).

Using the resulting main effects, an estimator for each run was calculated, and compared to the observed value. The large residual errors of observed figures minus estimated ones showed that the fitness of the sum model was inadequate (Table 3 and Fig. 3(a)).

### 3.1.3. Analysis Based on ‘Product Model’

The large variation of D data beyond four digits suggested that the D emission was not a sum but a product event of factors. We secondly tried to apply a product model to solve the data set.

The product model assumes the following data structure that each data \( x_i \) is expressed as the product of a base value \( c_0 \), multiplying factors \( \alpha_{ij} \), and an error \( e_k \).

\[ X_k = C_0 \cdot \prod \alpha_{i,j,k} \cdot e_k \] ..........................(4)

\[ \alpha_{i0} = 1, \quad \alpha_{i1} = \alpha_i \] ..........................(5)

where \( X_k \): observed data for Run \( k \) \((k = 1 - 8)\)
\( C_0 \): base with all levels = 0
\( \alpha_{ij} \): multiplying coefficient for factor \( i \), level \( j \) \((i = 1 - 7, j = 0 \text{ or } 1)\)
\( e_k \): error accompanied with Run \( k \)

The logarithm expression for Eq. (4) becomes

\[ \log X_k = \log C_0 + \sum \log \alpha_{i,j,k} + \log e_k \] ..........................(6)

The algorithm similar to sum model can give solutions to multiplying factors in Eq. (6).

The analysis based on the product model gave another set of results (Fig. 4), which proved that the product model was more reliable by less residual errors comparing to those based on the sum model (Table 3 and Fig. 2(b)).

Such analysis lead to the finding that EP dust, BF dust and scale purchased accelerated D emission significantly whereas quick lime suppressed it though the effect was in the same order as error. The effect of anthracite was insignificant. Domestic scale had less effect than scale purchased probably owing to its less contamination with oil and copper (Table 7).

### 3.2. Test 2

Since D data in Test 2 ranged narrowly from 0.068 to 0.37 ng-TEQ/Nm$^3$ (Table 2), the sum model was applied to calculate main effects, which had another advantage to estimate errors in familiar form (Table 4).

The increase of gas velocity, the decrease in coke content and turning off of after-burner were all D positive. Still the
phenomena were out of explanation. Enlargement in bed height reasonably increased D because of its increasing concentration of components beneath the frame front. Note, however, that these main effects were small comparing to the experimental error of 0.25 and 0.08 ng-TEQ/Nm³ appearing at the extra two lows in orthogonal array.

3.3. Multiple Regression Analysis of Commercial Plant Data

The multiple regression analysis brought the following findings. EP dust, scale purchased and BF dust gave positive effect on D emission; quick lime showed suppressing effect; the effects of domestic scale and anthracite were insignificant (Table 5). The result completely accorded with that obtained by the pot test 1 described in the Sec. 3.1. Of operational conditions, hearth layer appeared D-positive, but bed height turned insignificant. The effect of BTP for conventional machines remained hidden.

Since the residual errors of observed minus estimated were still as large as 1.0 ng-TEQ/Nm³ (Fig. 5), the multiple regression model obtained was inadequate for the practical use to control D emission within the regulation of 1.0 ng-TEQ/Nm³ for the existing machines. In other words, it would be difficult to control D emission just by controlling admixing amounts of revert materials. The poor estimation on the effect of BTP seemed to reduce the validity of the multiple regression model.

4. Discussion

4.1. Accuracy of D Data Observed

JIS K0311 requires more than four hours gas sampling time or more than 3 m³ volumes to ensure the accuracy of D data within 0.05 ng-TEQ/Nm³. As pot tests in this study lasted for 30–40 min, the sample gas accumulated to 0.3–0.6 m³, which was one tenth to one fifth of the manual-written volume. However, an analytical agency unofficially commented that they could guarantee one tenth of the nominal accuracy in case of JIS K0311 and could control their determination error within 0.1 ng-TEQ/Nm³ even in the shortage of sampling gas like the pot test.

The fact was that the standard deviation of error for D measurement exceeded the above figure, and reached to 0.2 ng-TEQ/Nm³ both for pot test and for plant operation (Table 6). This meant that the error depended on sintering process more than on D determination operation, and that 0.3–0.6 m³ sampling gas for pot test was enough to ensure the accuracy of D measurement.

4.2. D Formation Process in Sintering Process

According to Kasai, D formation in sintering process follows the 'de-novo' synthesis and soot-like carbon generated by partial oxidation of coke is chlorinated to be D with catalysis of copper. Nakano found by quenching pot test that the generation site was 1–3 cm below from the flame front in drying zone of around 400°C in temperature, where copper and chloride concentrated to significant extent which were supposed to have vaporized in melting zone.

**Table 4.** Comparison between D observed and estimated based on sum and product models.

| Factors       | Levels | Change of DXNs observed (ng-TEQ/Nm³) |
|---------------|--------|-------------------------------------|
| Gas velocity  | 0.6 → 0.85Nm³/s | +0.13 |
| Coke content  | 4.2 → 4.8%       | +0.105 |
| Bed height    | 400 → 600mm      | +0.26 |
| After burner  | On → Off         | +0.20 |
| EP dust       | 0 → 0.05%        | +0.09 |
| Errors        | (third column)    | -0.25 |
|               | (fifth column)    | +0.08 |

**Table 5.** Main effects of operational factors with EP dust and errors in Test 2.

| Ingredients       | A    | B    |
|-------------------|------|------|
| Machine number    | 1    | 6    |
| Sample number     | 27   | 30   |
| Scale purchased   | 0.31 | in   |
| Scale domestic    | in   | in   |
| EP dust           | 0.42 | ng   |
| BF dust           | 0.26 | in   |
| Anthracite        | in   | ng   |
| Quick lime        | -0.44|
| Bed height        | in   | in   |
| Hearth layer      | 0.46 | 0.21 |
| BTP               | 0.32 | in   |
| r                 | 0.71 | 0.70 |

Unit: Dioxins (ng-TEQ/Nm³)

in: insignificant
ng: neglected as showing the reverse effect than expected

**Table 6.** Factors investigated and multiple regression coefficients for (A) exhaust gas recirculation machine and (B) conventional machines.

| Factors          | Repeated data (ng-TEQ/Nm³) | Standard deviation | Coefficient of variation |
|------------------|--------------------------|--------------------|--------------------------|
| Plant            | 0.83, 0.98, 1.2          | 0.19               | 10%                      |
| Pot 1            | 1.1, 1.2, 0.97           | 0.12               | 11%                      |
| Pot 2 *)         | 1.7, 2.0                 | 0.21               | 11%                      |
| Test 2           | This work: estimated based on the third and fifth columns in Test 2 | 0.50               |

*) unpublished data

Fig. 5. Validity of the multiple regression models for (a) exhaust gas recirculation machine and (b) conventional machine.
According to Addink’s review, \(\text{de-novo}\) \(D\) synthesis gets vigorous between 300–400°C and needs 2–4 h to yield \(D\) to a significant amount. The relatively low temperature of reaction suggests the rate-determine step to be not mass transfer but chemical reaction, which is generally expressed as

\[
R = k[A]^a[B]^b[C]^c \quad \text{..........................(7)}
\]

\(k\): rate constant
\(A, B, C\): concentration of reactants
\(a, b, c\): order of reaction

\(D\) is composed from soot-like hydrocarbon (HC) and chlorine (Cl) with copper catalysis (Cu). Such elements derive from \(D\)-accelerating ingredients. Thus, the \(D\) concentration \((D_c)\) can be expressed as a product of causal ingredients.

\[
D_c = [\text{HC}] [\text{Cl}] [\text{Cu}] \quad \text{..........................(8)}
\]

Here, the factors \(a, b, c\) is arbitrarily simplified to be unity in Eq. (8).

Figure 3 presents evidence that \(D\) emission was a product event of causal ingredients, which basically agree with the Eq. (8).

### 4.3. Effect of Ingredients

Complicated was the formation route, the primary fact was that \(D\) needed Cl as an element and Cu as a catalysis, both of which existed in the ingredients of sintering mixture and that those containing hydro-carbon possibly fueled the \(D\) formation. Such fact enabled to estimate potential \(D\) accelerators from the chemical point of view (Table 7). BF dust was suspicious as it had chloride-coated carbon; scale was contaminated with oil and purchased one contained copper as well as oil; EP dust contained high Cl plus \(D\) itself; anthracite were doubted owing to high VM content. As a matter of fact, this research confirmed that all of them except anthracite had positive effect on \(D\) emission by both of pot test and plant data analysis.

| Ingredient | Ratio (mass%) | \(\text{D} (\text{ng/kg})\) | \(\text{Cl} (\text{mass%})\) | \(\text{C-H} (\text{mass%})\) | \(\text{Cu} (\text{mass%})\) |
|------------|---------------|-----------------|-----------------|-----------------|-----------------|
| EP dust    | 0.05-0.5      | 11-100          | 2               | 4.9 as Carbon   | 0.027           |
| Scale domestic | 0-3      | 1.9             | 0.01            | 0.01 as Oil     | n.d.            |
| Scale purchased | 0-3      | 0.02            | 0.4             | 0.24 as Oil     | 0.204           |
| BF dust    | 0-1           | 0.07            | 35 as Carbon    | n.d.            |
| Coke      | 3             | 1.3             | 0.11            | n.d.            |
| Anthracite | 0-1.5         | 0.1             | 5-6 as VM       | n.d.            |
| Return fines | 20        | 0.08            | 0.002           | -               |
| Orea      | 63            | 0.006           | 0.01-0.004      | -               |
| Lime stone| 13            | 0.006           | -               | -               |

n.d.: not detectible

### 2) Anthracite

Gevert et al.\(^7\) found a correlation between volatile organic compounds (VOCs) and \(D\) in exhausted gas, adding that the VOC originated from oil adhering on scale or that contained in BF dust due to PC/heavy oil injection. Fisher et al.\(^9\) confirmed the correlation based on their own data. Carbonaceous materials also emit VOC familiar as volatile matter (VM). Kawaguchi et al.\(^10\) intensively surveyed on various solid fuels, however, failed to obtain any tangible evidence proving anthracite \(D\)-positive. This work failed, too. VM from coal contains not only hydrocarbons but also NH\(_3\) that acts as a suppressor. The NH\(_3\) may cover hydrocarbon’s positive effect and allow anthracite to be \(D\)-negative.

### 3) Quick Lime

Quick lime was introduced as an economical binder to increase sintering productivity. This work reviled that the quick lime behaved as a \(D\)-suppressing agent. For the information, Bonte et al.\(^11\) had used it as an agent injected to main flue before de-duster.

Nakano et al.\(^12\) related that chlorine migrates to the reaction zone in the form of HCl (g). Chlorination of hydrocarbons generally occurs by Cl\(_2\) attacking, which generated

### Table 7. Cl, hydrocarbon, Cu and D contents of revert materials suspicious for \(D\) accelerator.

| Ingredient | Ratio (mass%) | \(\text{D} (\text{ng/kg})\) | \(\text{Cl} (\text{mass%})\) | \(\text{C-H} (\text{mass%})\) | \(\text{Cu} (\text{mass%})\) |
|------------|---------------|-----------------|-----------------|-----------------|-----------------|
| EP dust    | 0.05-0.5      | 11-100          | 2               | 4.9 as Carbon   | 0.027           |
| Scale domestic | 0-3      | 1.9             | 0.01            | 0.01 as Oil     | n.d.            |
| Scale purchased | 0-3      | 0.02            | 0.4             | 0.24 as Oil     | 0.204           |
| BF dust    | 0-1           | 0.07            | 35 as Carbon    | n.d.            |
| Coke      | 3             | 1.3             | 0.11            | n.d.            |
| Anthracite | 0-1.5         | 0.1             | 5-6 as VM       | n.d.            |
| Return fines | 20        | 0.08            | 0.002           | -               |
| Orea      | 63            | 0.006           | 0.01-0.004      | -               |
| Lime stone| 13            | 0.006           | -               | -               |

n.d.: not detectible

### Table 8. Effects of ingredients on \(D\) emission cited from Fig. 3(c) by Kasai et al.\(^7\)

| Ingredient | Ratio (g/kg mixture) | \(\text{PCDD/Fa} (\mu \text{g/kg mixture})\) |
|------------|----------------------|---------------------------------|
| Anthracite | 3%                   | 0.16                            |
| Scale      | 5%                   | 0.083                           |
| EP dust    | 1%                   | 0.1                             |
| BF dust    | 3%                   | 0.09                            |

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under the Deacon reaction: \( \text{HCl} + \text{O}_2 \rightarrow \text{Cl}_2 + \text{H}_2\text{O} \). Lime reduces the chlorination atmosphere by fixing HCl to CaCl\(_2\) that has the smallest Cl vapor pressure among metallic chloride. Such course of lime function could give the suppressing effect to quick lime.

### 4.4. Effect of Operating Condition

Though Putz et al.\(^6\) found a correlation of D emission to productivity, this work failed to obtain definite tendencies due to basic operational conditions including productivity beyond experimental error. Operational conditions can be paid less attention than the use of revert materials.

### 4.5. Possibility of Another Synthetic Site

Kasama et al.\(^{12}\) pointed out that a wind box (WB) was another niche for D synthesis, showing that D concentration peaked twice based on strand-ward measuring at every wind leg (WL) of Oita-IDL. They estimated that the first peak was due to D releasing that formed in bed, the last one was due to D synthesis at the WB. Actually, heated around 300–400°C, WB is dirty with seal grease and dusts bearing Cl, Cu, or soot-like substance, being suitable for de-novo synthesis.

After the multiple regression analysis failed to give a significant coefficient to BTP position as shown above, we tried a single regression analysis, plotting D data from a single machine on its BTP, and found a clear, negative correlation between BTP and D (Fig. 6) despite of plotting for the other machines in vain. The poor correlation seemed due to a hypothetical behavior of BTP that the machine overdriving beyond some critical speed held BTP indication to the ceiling. Though all discussion so far stands on an assumption that D synthetic site is in the sintering bed, the assumption cannot explain the BTP-D relationship. It should be understandable if some D synthesizes at WBs; that is, BTP backward movement can reduces the area of WBs synthesizing D, resulting in the decrease of D emission.

SDD committee pot-tested to find that hearth layer was another site.\(^{11}\) And the regression analysis in this work agreed with it. The main cause in hearth layer can be the dust that flies in from combustion zone. Which flies out as EP dust that is proved already to be a D accelerator.

### 5. Conclusions

Factors affecting D emission from sintering machine were investigated by means of pot tests and multiple regression analysis on commercial plant data. The findings were:

1. EP dust, BF dust and purchased scale were major D-accelerators; quick lime was D-suppressor. The D emission was a product event of each contribution: The multiplying coefficients were 28, 4, 4 and 0.7 times for 5 mass% EP dust, 5 mass% BF dust, 5 mass% purchased scale and 2 mass% quick lime, respectively.

2. The influence of operational condition: bed height, coke content, gas velocity, after burning, were insignificant.

3. WB can be another synthesis site as well as hearth layer.

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