ASSESSMENT CORRELATION OF TOTAL SUSPENDED SOLIDS (TSS) BASED ON DRIED AND SENSOR METHOD

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ABSTRACT: In order to maintain water quality standard due to mining activity, a very strict control is required on the water coming out into environment. In the field, water quality is assessed by using a portable Total Suspended Solids (TSS) measuring device that results are usually different from laboratory test results. Laboratory test produces accurate TSS values, but requires considerable time. Based on these conditions, it is necessary to study the comprehensive correlation between TSS results measured in the field by using portable device and the results by laboratory test. This study was conducted to determine correction of TSS measurement in the field by correlating it to the measurement in the laboratory. The same samples were used for both measurement methods and measured in unit of mg/l. Correction of TSS measurement using portable device was obtained by building a model of the relationship between TSS by device with TSS by laboratory test. Regression method was applied to obtain the calibration model. There were two portable devices using in this study, namely Partech 740 and DR900, so two models were built. Candidates for the models were constructed based on the grouping of relative error data resulted by each device. The models with the smallest mean absolute error (MAE) were selected as relatively fair model, which were TSS_{lab} = 0.0435 TSS_{Partech 740} + 66.86 for Partech 740 and TSS_{lab} = 0.6116 TSS_{DR900} + 85.46 for DR900, with MAE value of 30.38 and 30.92 respectively.

Keywords: TSS, Dried method, Relative error, Regression

1. INTRODUCTION

Mining activity, especially open-pit mining, has potential to increase value of Total Suspended Solids (TSS). However, if it is managed properly, water quality standard would be reached before the water is released into the environment. In order to ensure that the water has quality standard complying with law and regulation, continuous monitoring is required. TSS is measured by using a portable TSS meter in field and periodically tested in laboratory. The TSS values obtained from field measurement are usually not the same as the value from laboratory test. For this reason, a study is required to examine the correlation between TSS measured in field by portable device and TSS tested in laboratory, so that it may be used as a reference in controlling field activity.

Sand, silt, mineral precipitates, biological matter, and clay may be found in TSS. The clay is generally found as illite, kaolinite, or montmorillonite [1]. TSS is mainly formed due to physical processes related to hydrology, such as scouring of the river flow, erosion of adjacent surface soils and river banks, and aggregation of dissolved organic matter or chemical precipitation from inorganic solids in the water column [2]. TSS sample must represent the water body that is being tested and have representative sub-sample. In order to obtain representative sample of discharge that will be entering the water body from construction sites and utility pits, the samples should be collected at the end of the pipe [3]. A representative sample is required to be able to explain the condition of the overall water body.

TSS may be measured by a TSS meter/sensor, typically in unit of mg/L or ppm. TSS meter has a probe that must be connected to the TSS meter before putting it into a water sample. The probe will analyze the water sample and the TSS value, then, will appear on the display. TSS measurement by using sensor is relatively faster than the measurement in laboratory. In laboratory, TSS is measured by filtering out a water sample using a 47 mm glass fiber filter to separate the suspended solids from the water sample, drying the filter that has the solids on it, and then weighing it to determine the Total Non-filterable Residue (TNR) of the sample. This procedure is called the laboratory gravimetric procedure. TSS (in mg/L) is calculated as in

\[ TSS = \frac{(W_{fss} - W_f)}{V_s} \]  

where \( W_{fss} \) is weight of filter with suspended solids, \( W_f \) is weight of the filter, and \( V_s \) is volume of sample.

TSS measurement in laboratory takes about 2
hours or more for its entire process. It is not suitable for instantaneous and continuous measurement. The method of laboratory test refers to ASTM D5907 [4], EPA Method 160.2 [5], Standard Methods 2540D [6], or similar gravimetric method. For individual samples in time or space, a non-single ratio is required to obtain a reliable predictor of Total Dissolved Solids (TDS) based on TSS, or vice versa, due to their linear relationship that is not constant over time or space [7]. Suspended solids concentration is often estimated by measuring TSS as its primary parameter. For the alternative way, Suspended Solids Concentration (SSC) test may be conducted to estimate the suspended solids [8].

No measurement can be completely free of uncertainty, including uncertainty in scientific measurement that is called error. Errors in scientific measurement are inevitable and cannot be eliminated, so the best that may be done is to get assurance that the errors are as reasonably small as possible and to obtain a reliable estimation of how large the errors are [9]. In order to achieve consistency of measurement, it is fundamental to carry out calibration. One of the popular statistical methods in calibration is linear regression that is carried out by building a relationship model of a measurement value and reference value [10].

A research comparing TSS measurement by a sensor with the measurement by a laboratory test has been conducted. The result showed that sensor-reported result had comparable precision to the laboratory-analyzed result, while the accuracy was significantly different. The laboratory analysis gave more accurate results than the sensor did. Nevertheless, the sensor might be able to be used as initial testing to determine whether water samples required to be tested in the laboratory [11]. TSS and TDS have linear correlation, but the number of correlations is specific on each location [12]. During the first events, solid treatment efficiency for specific polluting load was increasing and then it was constantly decreasing [13].

2. RESEARCH METHOD

Sampling was carried out in two different locations with 15 samples taken in each location. Samples were taken at mine dewatering pump outlets at different time. Each sample was divided into 3 parts for testing. Water quality test was executed on pH, Fe, Mn and Cd as the parameters. These parameters refer to government regulation related to water management quality for mining activity. The laboratory test result can be seen in Table 1. Minimum and maximum values of the parameters based on laboratory test were applied as limitation for this research.

| Parameter | pH | Fe | Mn | Cd |
|-----------|----|----|----|----|
| Min       | 12.33 | 0.046 | 0.012 | 0.008 |
| Max       | 16.10 | 3.555 | 2.370 | 0.034 |
| Average   | 14.33 | 0.499 | 0.291 | 0.029 |

TSS was measured by three methods that are Partech, DR900, and laboratory test. There are many portable TSS meters equipped by sensor and probe that have been developed for instantaneous measurement, but Partech 740 and DR900 are the portable TSS meter used for this research among others. Measurement of TSS by using portable devices was carried out by inserting a sensor into a glass containing sample of water that would be measured in TSS for two different TSS meters (Fig.1).

Fig.1 Field measurement of TSS by using portable device (Partech 740)

The portable TSS measuring devices have an accuracy of 0.1 mg/L and the probe has an operating concentration range of 0 – 20,000 mg/L for Partech 740 and 0 – 750 mg/L for DR900. TSS measurement in the laboratory was carried out by using gravimetric method referring to [5,6,14]. A known volume of a water sample was put into a 47 mm pre-weighed glass fiber filter to separate the suspended solid from the water sample. The filter that had the solid on it, then, was dried by heated to constant mass at 104 ± 1 °C and weighed to determine the TNR. The mass increase divided by the water volume filtered is equal to the TSS in mg/L.

Referring to [6], the laboratory method was carried out by stirring a water sample with a magnetic stirrer, then a measured volume of the water sample was pipetted into the filtration apparatus by adjusting the total volume of water sample filtered should be at 2.5 – 200 mg of residue on the filter paper. After that, the filter was washed with three 10 mL successive washes, dried for one
hour at 103 to 105°C, cooled in a desiccator, and then weighed. TSS concentration was calculated by dividing the weight retained on the filter paper by the volume of sample filtered.

Measurement of TSS based on laboratory test is believed to be more accurate than using sensor. For this reason, in this analysis, TSS value by laboratory test was considered to be actual value. Both accuracy and precision of the measurement by devices can be calculated through relative error which is the difference in the value by sensors (measured value) and the value by laboratory test (actual value) compared to the value by laboratory test as the actual value (Eq. 2).

Relative error = \frac{|\text{Measured} - \text{Actual}|}{\text{Actual}} \quad (2)

The more similar the value by sensor with the value by laboratory test, the smaller the relative error, or in other words, the more accurate the sensor. Percent error which is the relative error multiplied by 100% shows how far the error resulted by the sensors is compared to the actual value. Precision of the measurement can be known by comparing standard deviation of the relative error to its mean.

Error pattern resulted by either Partech 740 or DR900 was further known through summary statistics and histogram. TSS data by each sensor was grouped into three based on the deviation from the average as shown in Fig.2.

Calibration was performed by using regression method on each group of data that had been formed to estimate the pattern of relationship between TSS value by each sensor and TSS value by laboratory test. This relationship can be used to estimate TSS value by laboratory test based on the value from the sensors by Eq.3.

\[ y = a + bx \quad (3) \]

where \( y \) is actual value (from laboratory test) and \( x \) is measured value (from sensor). \( a \) is a constant, while \( b \) is a coefficient of regression.

### 3. RESULT AND DISCUSSION

Table 2 shows the relative error of Partech 740 and DR900. The relative error of DR900 was much smaller than that of Partech 740; it means that DR900 was more accurate inasmuch as it was much closer than Partech 740 to the value by laboratory test. From Table 2, it can also be seen that the variation in the relative error of each sensor was not too large.

| Sample | Relative error of Partech 740 | Relative error of DR900 |
|--------|-------------------------------|-------------------------|
| 1      | 19.48                         | 0.124                   |
| 2      | 18.4                          | 0.136                   |
| 3      | 19.19                         | 0.33                    |
| 4      | 20.65                         | 0.52                    |
| 5      | 20.61                         | 0.477                   |
| 6      | 22.3                          | 0.543                   |
| 7      | 21.23                         | 0.566                   |
| 8      | 18.28                         | 0.416                   |
| 9      | 19.08                         | 0.448                   |
| 10     | 18.66                         | 0.397                   |
| 11     | 19.53                         | 0.499                   |
| 12     | 21.23                         | 0.682                   |
| 13     | 19.99                         | 0.412                   |
| 14     | 19.37                         | 0.381                   |
| 15     | 18.79                         | 0.389                   |
| 16     | 15.29                         | 0.004                   |
| 17     | 15.51                         | 0.148                   |
| 18     | 16.62                         | 0.447                   |
| 19     | 18.35                         | 0.232                   |
| 20     | 13.58                         | 0.289                   |
| 21     | 17.59                         | 0.391                   |
| 22     | 16.15                         | 0.059                   |
| 23     | 16.68                         | 0.155                   |
| 24     | 16.82                         | 0.238                   |
| 25     | 17.51                         | 0.257                   |
| 26     | 17.59                         | 0.314                   |
| 27     | 17.97                         | 0.15                    |
| 28     | 16.57                         | 0.191                   |
| 29     | 16.83                         | 0.174                   |
| 30     | 14.4                          | 0.022                   |

Fig.2 Grouping data under a normal curve that lie between 1, 2, and 3 standard deviation on each side of the mean.
In order to find out more about the relative error of both sensor, summary statistics (Table 3) and histograms (Fig.3) were made.

Table 3 Summary statistics of relative error for Partech 740 and DR900

|                | Relative error of Partech 740 | Relative error of DR900 |
|----------------|-------------------------------|-------------------------|
| Mean           | 18.14                         | 0.313                   |
| Variance       | 4.33                          | 0.030                   |
| Std. deviation | 2.08                          | 0.174                   |
| Min. value     | 13.58                         | 22.30                   |
| Max. value     | 0.004                         | 0.682                   |

The averages of relative error for Partech 740 and DR900 were significantly different that were 18.14 and 0.313, as shown in Table 2. It means that TSS value by Partech 740 deviated on average of 1,814% from the actual value – very far from the actual value compared with TSS value by DR900 that deviated on average of 31.3% from the actual value. Therefore, DR900 was more accurate than Partech 740 due to its mean of relative error which was much smaller. However, Partech 740 appeared to be more precise than DR900, known from its standard deviation compared to its mean which was smaller than that of DR900. Variability of relative error for Partech 740 ranges from 13.58 to 22.30, while for DR900, it ranges from 0.004 to 0.682. Visually, the variability can be seen from the narrowness of normality curve as in Fig.3.

Fig.3 shows histograms of relative error for Partech 740 and DR900 with an addition of normality curve. Both data followed normal distribution according to the Shapiro-Wilk normality test with a significance level of 5% that resulted p-values of 0.994 and 0.691 respectively for Partech 740 and DR900. Partech 740, which had previously been described as more precise than DR900, had a narrower curve than the DR900.

By considering the relative error data distribution, three types of calibration models for each sensor were constructed: involving 100% data, 95% data, and 68% data. The results are presented in Fig.4 for Partech 740, and Fig.5 for DR900.

Fig.4 Plot of TSS value by laboratory test against TSS value by Partech 740 as well as the excluded data points on model 95% (red) and 68% (yellow)

When considering 95% data of relative error, only 1 sample was excluded for both Partech 740 and DR900, which were sample no. 20 for Partech 740 and sample no. 12 for DR900. The excluded sample for each sensor had value of relative error that was the farthest from average. In both Fig.4 and Fig.5, the excluded sample for group that considered 95% data of relative error was presented in red.

When considering 68% data of relative error, more samples were excluded. There were 9 samples excluded for Partech 740, which were samples no. 4, 5, 6, 7, 12, 16, 17, 20, and 30. For DR900, the excluded samples were no. 1, 2, 4, 6, 7, 11, 12, 16, 22, and 30. In both Fig.4 and Fig.5, the samples were in yellow.
Fig. 5 Plot of TSS value by laboratory test against TSS value by DR900 as well as the excluded data points on model 95% (red) and 68% (yellow)

For each model constructed, Mean Absolute Error (MAE) was calculated. For each sensor, the model with lowest MAE among all was chosen as a calibration model that was considered to be relatively fair. The second model, either for Partech 740 or DR900, had the minimum MAE. Therefore, for both sensors, the second model, which was a model with the data of relative error included in the interval of 95%, was chosen (Fig. 6).

Based on the explanation above, TSS resulted from each sensor had correlation with TSS from laboratory test that were different from each other. Also, each sensor showed different calculation number to calculate the same sample.

4. CONCLUSION

Measuring Total suspended solid (TSS) using sensor is shown accurate but it’s can be applied on field before sample sent to the laboratory. TSS value measured by sensors, which are Partech 740 and DR900, can be converted to approximate the actual value (TSS by laboratory test) through the following equations:

\[
TSS_{\text{Lab}} = 0.0435 \times TSS_{\text{Partech}} + 66.86 \\
TSS_{\text{Lab}} = 0.6116 \times TSS_{\text{DR900}} + 85.46
\]

However, it should be noted that the average deviation from the actual value, expressed with MAE, produced by the two models are 30.38 and 30.92. Completely accurate calibration model is difficult to construct due to the random errors. It also should be noted that these formula are just applicable for water quality that has parameters stated in Table 1.

Further testing is required to confirm the result of the analysis due to the minimal amount of sample used in this analysis. Detailing water quality is also recommended to guideline application the correlation formula has been established above.

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