# QUALITATIVE METHODS FOR DETECTING THE SEDIMENT SOURCES BY GRAIN SIZE DISTRIBUTION AND X-RAY DIFFRACTION

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The sources of sediment deposited in Sengguruh and Sutami reservoirs are detected by qualitative analysis of grain size distribution and mineral composition resulted from x-ray powder diffraction (XRD). Grain size distribution of sediment in Sengguruh reservoir shows that Lesti sub-basin contributes higher percentage of sand particles than Brantas sub-basin. The most dominant minerals in Sengguruh reservoir are Feldspar and Carbonate (Calcite). In Sutami reservoir, the most dominant minerals are Feldspar, Mica and Chlorite. The difference of mineral composition both of reservoirs indicates the increasing of sedimentation from Metro-sub-basin. Now, it should be considered while it was neglected in the past.

Finally, the proposed method is applicable to assess sub-basins contribution of sediment origin and to evaluate the existing sedimentation countermeasures.

Key Words : qualitative, sediment source, grain size, mineralogy, x-ray diffraction

# **1. INTRODUCTION**

Characteristic of reservoir sediments is an important point to identify the part of sub-basin or the source from which material comes from, especially in the case of basin which the surface erosion plays a key role as a dominant process for sediment accumulation.

The material we commonly refer as reservoir sediment is soil which usually composed of solid particles, liquid and gas with wide range from very soft, organic deposited through less compressible clays and sands to soft rock. The solid phase of a soil may content various amount of crystalline clay and non clay minerals<sup>1)</sup>.

Identification of the minerals, both clay and non clay, can ordinarily be made by using X-ray diffraction. Grain size distribution, other physical properties and simple chemical test is used to indicate the presence of organic matter and other constituents<sup>2)</sup>.

Sengguruh and Sutami reservoirs, located in upper-part of Brantas basin-Indonesia, are suffering from sedimentation. Many researchers are interested to identify the origin of sediment yields. Osanai  $et.al^{3}$  is trying to identify the origin of sediment using isotope tracer. Sayama  $et.al^{4}$  did numerical simulation for assessing sediment fields. Nakagawa  $et.al^{5}$  analyzed the remote sensing data and field observation to identify sediment yield and the transport processes.

In order to get characteristics of sediment deposited in reservoirs, detecting the sediment sources and support previous researches of quantitative analysis, the sediment characterization using grain size distribution, specific gravity, atterberg limits, chemical properties and X-ray diffraction analysis was done in this study.

By qualitative analysis, result found shows big contribution of Metro-sub basin in Sutami reservoir. In previous studies, Metro sub-basin has been neglected both by researchers and authority. Moreover, field investigation shows the landuse in Metro sub-basin has been extensively changed. Furthermore, it gives more valuable information for following researchers to evaluate the existing countermeasures.

## 2. SAMPLING MATERIAL

The sources of sediment were collected from surface soils and bedload material of sub-basins where susceptible to erosion (Brantas, Lesti and Metro)<sup>6,7)</sup>. Samples were taken from 3-5 locations of each sub-basin. The deposited sediment was collected from reservoirs by auger and core sampler. The sampling depth varied from 0.5 to  $5m^{80}$ .



Fig.1 Locations of material sampling



Fig.2 Locations of samples in Sengguruh reservoir (1-5m depth)

In Sengguruh reservoir, the samples were collected at each tributary (SL1, SB1) and pool (SL2, SB2) as shown in Fig. 2. In Sutami reservoir, they were collected at reservoir entrance (Dempok) and pool (ST1, ST2, ST3, ST4) as shown in Fig. 3.



Fig.3 Locations of samples in Sutami reservoir (0.5-1m depth)

## **3. METHODOLOGY**

## (1) Grain size Distribution

The grain size distribution of material sediment in the basin is one of important assessment of sediment origin beside of mineralogical content analysis. The major effects of transportation processes on the physical properties of sediment are sorting and abrasion. The sorting produces a progressive decrease in particle size with distance from the sources. In general, abrasion changes the size of particle into smaller and also changes the shape into round. Theoretically, sediment sorting will be useful as quick information to identify the source of the sediment comes from.

## (2) Other Sediment Properties

Additional sediment properties such as specific gravity, atterberg limits and chemical properties were analyzed to get more comprehensive results.

## (3) Mineralogy Analysis

X-rays are electromagnetic radiation with a wavelength of about  $1\text{\AA}(1^{-10} \text{ m})$ , which is about the size of an atom. X-ray diffraction is one of the most important characterization tools used in solid state materials science.

Mineralogical decomposition of the deposited sediment is described by X-Ray Powder Diffraction (XRD). XRD is useful for identifying fine grained minerals that may not be analyzed by other techniques. Mineralogical decomposition analysis using XRD is shown in **Fig. 4**.

When X-ray beam hits a sample, it is diffracted (**Fig. 5**). The distances between atoms is calculated by applying the Bragg's Law,  $n\lambda=2dsin\theta$ , where *n* is the order of diffraction,  $\lambda$  is wavelength of incident x-ray beam, *d* is distances between adjacent atoms (*d*-spacing), and  $\theta$  is the angle of incidence of x-ray beam. The characteristic of *d*-spacing generated in a typical X-ray scan provided fingerprint of the mineral present in the sample.



Fig.4 Processing X-ray powder diffraction for mineralogy decomposition



Fig.5 Schematic of an X-ray powder diffractometer

Table 1	. Instrumentation	of X-ray	UV Diffraction <sup>8)</sup>
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X-RAY SPECIFICATIONS					
Diffractometer type	PW3710 BASED				
Tube anode	Cu				
Generator tension [kV]	40				
Generator current [mA]	30				
Wavelength Alpha-1 [Å]	1.54056				
Wavelength Alpha-2 [Å]	1.54439				
Intensity ratio (Alpha-2/Alpha-1)	0.500				
Divergence slit [ <sup>o</sup> ]	1				
Receiving slit [ <sup>o</sup> ]	0.2				
Monochromator Used	NO				
Start angle [°20]	4.010				
End angle $[^{\circ}2\theta]$	59.990				
Step size [°2θ]	0.020				
Time per step [s]	0.100				
Type of scan	CONTINUOUS				

## (4) Analysis of X-ray Patterns

XRD complements other results to get better interpretation of sediment mineralogy. A complete x-ray diffraction pattern consists of a series of reflections of different intensities and values of  $\theta$ . XRD data can be analyzed to determine the proportion of different minerals present.

Using XRD diffraction patterns in **Fig. 6**, information used in the assessment of soil mineralogy are peak position, peak intensity and peak shape. The type of mineral is distinguished from peak position by recalling the Bragg's Law for specific mineral. Ratio of peak intensity of different order reflections is useful to qualitatively evaluate structure of mineral in the soil sample. It gives clue of the richness of minerals in the sample. The sharper peak-width indicates how well-crystalline of mineral elements are.



Fig.6 X-ray diffraction pattern of sediment sample

Extensive X-ray diffraction data for the clay minerals and other common soil minerals are listed by Moore and Reynolds in Mitchell *et al*<sup>2)</sup>.

## **3. RESULTS AND DISCUSSIONS**

## (1) Grain size distribution of sediment

Fig. 7 shows the comparison of grain size distribution of sediment samples from the sources (Brantas, Lesti and Metro) and deposited in Sengguruh (SL1, SL2, SB1, SB2) and Sutami reservoirs (Dempok, ST1, ST2, ST3, ST4).

In Sengguruh reservoir, Lesti sub-basin has higher percentage of sand particle than Brantas subbasin. This evidence shows that Brantas sub-basin has more surface erosion occurrences (gully, rill and inter-rill) or bank collapse, because this occurrence resulted higher rate of silt-clay particle. In a fact, there are extensive sand mining in upper part of Lesti basin and extensive bareland cultivation in upper part of Brantas basin.

Basically, sediment material was trapped in Sengguruh reservoir first before it was released into Sutami reservoir as suspended load. The grain size distribution of **ST1**, **ST2**, **ST3** and **ST4** has lower percentage of sand-particle due to regularly dredged at Dempok. However, sand particle in Dempok shows higher percentage of sand particle and has similar distribution with Metro sub-basin. Since there is no inflow other than Metro River, it indicates increasing contribution of coarser material from Metro sub-basin. This information is useful finding to evaluate the existing reservoir sedimentation countermeasures.



Fig.7 Percentages of sand and silt-clay mineral between deposited and sources material in Sengguruh (a) and Sutami (b) reservoirs



Fig.8 Comparison of grain size distribution of deposited material in Sengguruh and Sutami reservoirs



Fig. 9 Grain size distribution of sediment from Brantas and Lesti sub-basins and deposited in Sengguruh reservoir

Nowadays, many researchers do not consider the Metro sub-basin in Sutami sedimentation due to lack of information (as no observation equipments are installed) and/or assuming that no considerable landuse change has taken place. Now, Metro subbasin should be considered much in sedimentation countermeasures of Sutami reservoir, while existing countermeasures consider only from Brantas and Lesti sub-basins.

The other information is materials deposited in Sutami have significant difference in size comparing material deposited in Sengguruh reservoir because Sengguruh is sediment trap for Sutami, that's why order of sediment size of Sutami less than Sengguruh (**Fig. 8**). In general, the sediment sorting process shows sediment size in the upper part is coarser than lower part as shown in **Fig. 9**.

## (2) Other Sediment Properties

There is no much difference between the specific gravity of sediment (Gs) deposited in reservoirs and that of sources (**Table 2**). From **Table 2**, the value of Gs at Dempok shows lighter than any other values of Gs of Sutami reservoir (**ST1**, **ST2**, **ST3**, **ST4**). However, it was similar to Gs value of Metro sub-basin.

Since there were landuse changes in Metro subbasin, the surface erosion and riverbank collapse were occurred. This indicated that such kind of processes in Metro sub-basin have been affected Gs value at Dempok and showed the increasing of sediment contribution from Metro sub-basin. Otherwise, the value of Gs in Dempok should has similar value to any location of Sutami reservoir (ST1, ST2, ST3, ST4).

Table 2 Comparison of Specific Gravity (Gs) of sediment

Reservoir	Location	Gs	Reservoir	Location	Gs
Sengguruh	Brantas	2.01	Sutami	Metro	2.01
	SB2	2.29		Dempok	1.86
	SB1	2.37		ST1	2.45
	Lesti	2.17		ST2	2.44
	SL2	2.33		ST3	2.51
	SL1	2.31		ST4	2.52

Another physical properties of the deposited sediment such as Liquid Limit (LL) and Plastic Limit (PL) shows that sediment in Brantas reach contains micaceous or diatomaceous fine sandy, silty soils, elastic silts, organic silts, clays and silty clays (OH). In Lesti reach, it is mainly composed of inorganic and organic silts and silty clays with low plasticity, rock flour, silty or clayey fine sands (OL). The sediment in Sutami reservoir is categorized to be inorganic clays with high plasticity (MH), as shown in Table 3 and Fig. 10. Because of clay-soil is not 100% clay, based on its activity value of **IP** divided by percentage of clay particle<sup>9)</sup>, the associated mineral is roughly detected as Kaolinite in Sengguruh and Montmorillonite in Sutami reservoir. Detail description of fundamental mineral is interpreted from XRD results.

Table 3 Atterberg limits of deposited sediment

Reservoir	LL	PL	IP	Soil Class	
Sengguruh					
SL2	43.62	34.73	8.89	OL	
SB2	55.81	41.04	14.57	OH	
Sutami					
ST4	55.62	30.80	24.82	СН	
ST3	56.52	30.75	25.76	СН	



Fig.10 Comparison of Plasticity Index (IP) and Liquid Limit (LL) of deposited sediment in reservoirs

Table 4 Chemical properties of deposited sediment in reservoirs

	RESULTS							
Parameter	SB1	SB2	SL1	SL2	ST1	ST2	ST3	ST4
рН	8.15	7.30	8.07	8.06	7.09	7.10	7.16	7.44
CO <sub>3</sub> <sup>2</sup>	915.08	880.80	898.71	881.64	829.08	828.30	832.21	848.87
CI.	20.26	25.73	19.38	22.04	146.22	154.29	167.65	132.50
LOI	-	75.99	-	89.70	-	-	81.29	85.75
TOC	4.25	12.55	2.80	8.09	11.29	10.69	-	10.34
Total SO <sub>4</sub> <sup>2</sup>	0.06	0.23	0.11	0.17	0.03	0.08	0.03	0.17

LOI = Loss On Ignition - = not availlable

Based on the chemical properties analysis (**Table 4**), almost all of sediment samples have pH value around 7-8. Carbonate content of samples are 800 - 900 ppm. In average, sediments of Sengguruh reservoir are more alkaline than that of Sutami reservoir due to higher carbonate content.

LOI (Loss on Ignition) of **SL2** (Lesti) is lower than **SB2** (Brantas) and increase again in Sutami (**ST3, ST4**). Higher LOI value shows higher organic content in soil. TOC (Total Organic Carbon) value in **SL1** (Lesti) is the lowest. Higher value of TOC in deposited sediment shows higher surface erosion occurred in the basin area, because inorganic matters can dilute organic matters during surface erosion process<sup>10</sup>. Field investigation shows that the surface erosion of Brantas sub-basin is higher due to extensive cultivation in upstream of Brantas subbasin while in Lesti sub-basin decrease and change to extensive sand mining.

## (3) X-ray Powder Diffraction Results

Using XRD results, more detail information of mineral content in sediment deposited was obtained. The result of XRD is figured like **Fig. 11**. Other samples are done in same manners. From these figures mineral and it behaviors are identified. In **Fig. 12**, the mineral occurrence at Sengguruh reservoir consists of Feldspar, Carbonate (Calcite), Kaolinite, Mica, Chlorate, Montmorillonite, Illite and Quartz. Feldspar almost dominantly came from Lesti reach (**SL2**) and Carbonate (Calcite) from Brantas reach (**SB2**). From the peak width, the shaper is Feldspar, Carbonate (Calcite), Illite and Quartz. They are identified as well crystalline structure.

In Sutami reservoir, the mineral is dominated by Feldspar, Chlorite and Montmorillonite at the reservoir entrance and Feldspar, Mica and Chlorite at the main pool. The well crystalline structure is identified as Illite.

All of the XRD results are well agreeing with preliminary identification based on activity value (IP / % clay particle) in previous passage.

Feldspar is a group name of large number of aluminum silicate minerals of variable composition. The general formula is **X** Al(Al, Si)Si<sub>2</sub>O<sub>8</sub>, where **X** may be Na, K, Ca or Ba. These minerals are softer than Quartz and slightly reactive. Illite is widely distributed in nature, abundant, and often the dominant clay mineral in soil, sedimentary rocks, and freshwater sediments<sup>11</sup>.



Fig.11 Example analysis of XRD results for Sengguruh reservoir (SL2)



Fig.12 Major mineral in Sengguruh (left) and Sutami (right) reservoirs

The abundance of Feldspar in Sengguruh reservoir is related to natural processes such as weathering, chemical and transport process. The sand mining of both rivers, Brantas and Lesti, has contributed to the presence of feldspar, because of feldspar contains more silicate and most affected by sand occurrence.

In addition, Montmorillonite and Illite are softer and lighter then Feldspar and more reactive. Sediment will be deposited in Sengguruh first, hence softer and lighter minerals are easily released to downstream due to their natural characteristics and some influences has occurred on its way.

Moreover, in Sutami reservoir, the difference of mineral composition with Sengguruh reservoir is another clue to look at other environmental and subbasin contribution (Metro), because human activities is one of factors affected natural mineral occurrence.

# 4. CONCLUSION

The characteristics of deposited sediment in Sengguruh and Sutami reservoirs are identified as the following:

- 1. In Sengguruh reservoir, Brantas reach has finer fractions (silt-clay) than Lesti reach due to surface erosion occurred in Brantas sub-basin while in Lesti basin has more sand mining.
- 2. Coarser sediment is trapped in Sengguruh reservoir, while the finer is released down to Sutami reservoir as suspended load. However, sediment grain size in Dempok shows high percentage of sand particle than other locations of Sutami reservoir (ST1, ST2, ST3, ST4) and has similar distribution to Metro sub-basin. It indicates high contribution of coarser sediment coming from Metro basin. This information is useful finding to evaluate the existing reservoir sedimentation countermeasures. In the previous studies, researchers do not consider much of the Metro sub-basin contribution due to lack of information and/or assuming no considerable landuse changes.
- 3. From mineralogy view, Feldspar in Sengguruh reservoir is dominantly coming from Lesti reach

and Carbonate (Calcite) is coming from Brantas reach. In Sutami reservoir, the major mineral is dominated by Feldspar and Chlorite at the entrance. Feldspar, Mica and Chlorite are found at the main pool. The difference of mineral composition both of reservoirs indicates the increasing of sedimentation from Metro-subbasin.

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