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# CHARACTERIZATION OF COAL ASH

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## ABSTRACT

Coal ash samples were taken from Calaca, Thermal Power Plant in Batangas in 1993 and in 1996. These coal ash samples were screened into different particle sizes. Each particle size was analyzed for its moisture content, amount of unreacted carbon, mineral content and its fusibility. The effect of the different particle sizes on the ash fusibility was investigated. This is a sequel to a study conducted by Pugal, D.L. et al (1995). Results of the characterization show that sieving coal ash samples into different particle sizes give higher sintering, softening and melting temperatures. Big particle sizes also have higher sintering, softening and melting temperatures than the smaller particle sizes.

## INTRODUCTION

Coal-fired thermal power plants in the Philippines are envisioned to increase its capacity for the next 30 years from 1460 MW in 1996 to 10,120 MW in 2025. This increase in power generation capacity would mean increase utilization of both local and imported coal. It also means increase emissions of hazardous and toxic pollutants such as sulfur emissions as well as increase production of coal ash, the by-product obtained from burning coal. These environmental problems must be addressed properly in order to realize actual implementation of the Power Development Program of the Philippines which is designed to meet the future power requirements of the country as dictated by economic growth targets.

The ultimate objective of coal ash characterization is to produce a high quality absorbent for $SO_x$ through the addition of limestone. The method of production is an alternative to the energy intensive process which use calcined limestone. The process will utilize coal ash and raw limestone as the principal raw materials in a temperature-controlled curing vessel. Another important feature of the method of production will be the utilization of waste $CO_2$ for the polarization of Ca in $CaCO_3$, thus eliminating the calcination process.

The Industrial Technology Development Institute (ITDI) of the Department of Science and Technology (DOST) through its Fuels and Energy Division (FED) has entered into a joint collaboration with the Hokkaido National Industrial Research Institute (HNIRI), Agency of Industrial Science and Technology (AIST), Ministry of International Trade and Industry (MITI), Japan to conduct a research study on the preparation of highly active absorbent for $SO_2$ removal from coal ash.

At present, the Philippines has four (4) existing coal-fired thermal power plants located in Calaca, Batangas, Cebu and Pagbilao, Quezon with a total rated capacity of 1441 MW. These power plants utilizing blended sub-bituminous Semirara and Australian coal generate about 1 million metric ton of ash per year. At present, these plants do not have a means for controlling sulfur emissions. Adapting technologies such as the application of absorbent for $SO_2$ removal from coal ash could minimize the environmental impact of the operation of coal-fired power plants.

## MATERIALS AND METHODS

### Collection of Coal Ash Samples

Two sets of coal ash samples, one collected in 1993 and the other collected in 1996, were used in the experiments. These samples were collected from Calaca Thermal Power Plant in Batangas by pulverized coal combustion process. The 1993 coal ash samples used are the same samples which have been described earlier by Pugal, D.L. et al (1995).

### Particle Size Analysis

To determine the effect of particle size on the fusibility of coal ash, a particle size analysis was done. The 1993 and 1996 coal ash samples were screened using Tyler sieves of different screen sizes and Ro-tap machine. The particle sizes used and studied are tabulated in Table 1.

### X-ray Powder Diffraction

X-ray powder diffraction analysis was carried out to determine the mineral constituents of each particle size of the 1993 and 1996 coal ash samples. X-ray diffraction profiles were determined by the use of a Rigaku X-ray Diffractometer Model HV21 for the1993 coal ash samples and Rigaku Geigerflex for the 1996 coal ash samples.

### Scanning Electron Microscope

The microstructure of each particle size of the 1993 and 1996 coal ash samples was observed through a JEOL Scanning Electron Microscope Model JSM-TSO using an acceleration voltage of 20 kV. Through this microscope, the surface phenomena of the coal ash samples were observed.

## Thermal Analysis

Thermal analysis was carried out for each particle size of the 1993 and 1996 coal ash samples using a Rigaku Thermoflex unit with $\alpha$-$Al_2O_3$ as reference sample. The coal ash samples were heated up to 1500°C at a heating rate of 10°C /min under air atmosphere, TG range of 20 mg full scale and DTA range of $\pm 50\,\mu V$.

## Ash Fusibility Determination

Ash fusibility of each particle size of the 1993 and 1996 coal ash samples was determined using the thermal analysis data. From this data, the sintering temperature (SIT), softening temperature (SOT) and melting temperature (MT) were read.

# RESULTS AND DISCUSSION

## Particle Size Analysis

Table 2 shows the weight fraction of the different particle sizes for both the 1993 and 1996 coal ash samples with the corresponding moisture content and amount of unreacted carbon while Table 3 shows the cumulative weight fraction.

About 55.77% of the 1993 coal ash samples is smaller than 32 $\mu$m and 16.90% is smaller than 45 $\mu$m and bigger than 32 $\mu$m while 71.95% of the 1996 coal ash sample is smaller than 38 $\mu$m. This means that the coal ash being produced at Calaca Thermal Power Plant do not vary much in particle size.

To conduct further investigation on the extent of variation of the particle size, the distribution for both the 1993 and 1996 coal ash samples was plotted in Figure 1. From this figure, $Dp^{50}$ for the 1993 coal ash sample is 27 $\mu$m while $Dp^{50}$ for the 1996 coal ash sample is 24 $\mu$m. $Dp^{50}$ is the particle size wherein 50% of the sample passes.

As shown in Figures 2 and 3, the amount of unreacted carbon of each particle size of the 1993 and 1996 coal ash samples, respectively, was plotted against the weight fraction. For both 1993 and 1996 samples, a large amount of unreacted carbon can be found in particle sizes greater than 75 $\mu$m.

In Figures 4 and 5, the amount of unreacted carbon in relation to the cumulative weight fraction is shown. For the 1993 coal ash samples, the amount of unreacted carbon is more than 5% for particle sizes greater than 106 $\mu$m while for the 1996 coal ash samples, the amount of unreacted carbon is less than 5% for all particles sizes.

## X-ray Powder Diffraction

Figures 6 and 7 show the major mineral constituents present in each particle size of the 1993 and 1996 coal ash samples, respectively. Quartz, mullite, magnetite, calcite and lime are

the major minerals present in the 1993 coal ash samples while quartz, mullite, magnetite and monticellite are the major minerals present in the 1996 coal ash samples. Quartz have high intensity peaks in particles sizes larger than 75 $\mu$m for both the 1993 and 1996 coal ash samples while magnetite, monticellite and lime exhibit low intensity peaks.

## Scanning Electron Microscope

The microstructures of each particle size of the 1993 and 1996 coal ash samples are shown in Figures 8 and 9, respectively. As the particle size grows smaller, the shape of the particles becomes more regular and forms a balloon shape. A large percentage of the big particle sizes have irregular forms.

## Thermal Analysis and Ash Fusibility Determination

Figures 10 and 11 show the TG-DTA curves obtained for each particle size of the 1993 and 1996 coal ash samples, respectively. These curves depict the weight loss pattern and the different endothermic and exothermic reactions which occur during the heating of the coal ash samples.

From the DTA curves, it can be observed that in the temperature region just above 500°C, exothermic effects due to the oxidation of organic substances for all particle sizes occur. As the particle size grows larger, the exothermic peak becomes more prominent. This is because unreacted carbon is present in large quantities in big particle sizes.

The sintering, softening and melting temperatures were read from the TG-DTA curves and these are shown in Table 4. The 1993 coal ash samples have higher sintering (1120 -1230°C), softening (1193 -1288°C) and melting temperatures ( > 1500°C ) compared with the 1996 coal ash samples (1102 - 1117°C for sintering, 1160 - 1194°C for softening and 1450 -> 1500°C for melting temperature). High sintering, softening and melting temperatures were observed for samples with big particle sizes.

To determine the effect of sieving on the coal ash samples, the sintering, softening and melting temperature were obtained for a coal ash sample without sieving (NSCFA). The coal ash samples with sieving exhibit higher sintering, softening and melting temperatures that the coal ash samples without sieving. Hence, sieving results in higher ash fusibility.

# CONCLUSION

In the studies conducted, it has been established that particle size has an effect on the fusibility of coal ash. Coal ash having a particle size larger than 48 $\mu$m exhibits melting temperatures greater than 1500°C. These high melting temperatures in large particle sizes are attributed to the presence of high content of silicates (quartz, mullite and monticellite). In addition, large particle sizes contain considerable amounts of unreacted carbon.

Based on the thermal analysis and ash fusibility determination, sieving coal ash into different particle sizes gives higher sintering, softening and melting temperatures. Sieving coal ash separates the large particle sizes having high ash fusibility from the small particle sizes having lower ash fusibility.

This study stressed the importance of determining the physical, chemical and thermal characteristics of coal ash which is used as the starting raw material for the preparation of high quality absorbent for $SO_x$ in coal-fired thermal power plants. Results obtained from these characterization studies show that it is possible to predict the fusibility of coal ash given the chemicals and mineral composition of the coal ash.

## ACKNOWLEDGMENT

The authors wish to acknowledge the assistance of the staff of the Standards and Testing Division in the analysis of coal ash and Mr. Elvin M. Alcoba in the preparation of the illustrations.

## REFERENCES

1. Pugal, D.L. et al. Studies on the Fusibility of Coal Ash. Philippine Technology Journal, Vol. XX, No. 1, January-March 1995.
2. Philippine Energy Plan, 1996 - 2025, Department of Energy, Philippines.
3. Vassilev, S.V. et al (1995). Influence of Mineral and Chemical Composition of Coal Ashes on their Fusibility. Fuel processing Technology, Vol. 45.

**Table 1**
Particle Sizes Studied

| 1993 Samples | Particle Size | 1996 Samples | Particle Size |
|---|---|---|---|
| 93-1 | +250 μm | 96-1 | + 106 μm |
| 93-2 | 150 - 250 μm | 96-2 | 75 - 106 μm |
| 93-3 | 106 - 150 μm | 96-3 | 53 -75 μm |
| 93-4 | 75 - 106 μm | 96-4 | 48 -53 μm |
| 93-5 | 53 - 75 μm | 96-5 | 38 - 48 μm |
| 93-6 | 45 - 53 μm | 96-6 | - 38 μm |
| 93-7 | 32 - 45 μm | | |
| 93-8 | - 32 μm | | |

**Table 2**
Weight Fraction of the Different Particle Sizes

| Particle Size | Weight Fraction (wt%) | Proximate Analysis | |
|---|---|---|---|
| | | Moisture (wt%) | Unreacted Carbon (wt%) |
| 96-NSCFA | - | - | 3.10 |
| 96-1, +106 μm | 3.10 | 0.25 | 7.72 |
| 96-2, 75~106 μm | 5.20 | 0.36 | 5.88 |
| 96-3, 53~75 μm | 6.80 | 0.45 | 4.96 |
| 96-4, 48~53 μm | 4.75 | 0.58 | 3.32 |
| 96-5, 38~48 μm | 8.20 | - | 3.14 |
| 96-6, -38 μm | 71.95 | 0.09 | 2.84 |
| 93-NSCFA | - | - | 6.32 |
| 93-1, +250 μm | 1.16 | - | 40.34 |
| 93-2, 150~250 μm | 4.12 | - | 17.28 |
| 93-3, 106~150 μm | 4.33 | - | 14.67 |
| 93-4, 75~106 μm | 5.45 | - | 11.06 |
| 93-5, 53~75 μm | 6.09 | - | 9.01 |
| 93-6, 45 ~53 μm | 6.18 | - | 7.61 |
| 93-7, 32~45 μm | 16.90 | - | 5.61 |
| 93-8, -32 μm | 55.77 | - | 3.46 |

**Table 3**
Cumulative Weight Fraction

| Particle Size | Cumulative Weight Fraction (wt%) | Unreacted Carbon Weight Fraction (wt%) |
|---|---|---|
| 1996 Coal Ash | | |
| -38 μm | 0.7195 | 0.0284 |
| -48 μm | 0.8015 | 0.0287 |
| -53 μm | 0.8490 | 0.0290 |
| -75 μm | 0.9170 | 0.0305 |
| -106 μm | 0.9690 | 0.0320 |
| +106 μm | 1.0000 | 0.0334 |
| 1993 Coal Ash | | |
| -32 μm | 0.5577 | 0.0346 |
| -45 μm | 0.7267 | 0.0396 |
| -53 μm | 0.7885 | 0.0425 |
| -75 μm | 0.8494 | 0.0459 |
| -106 μm | 0.9039 | 0.0498 |
| -150 μm | 0.9472 | 0.0542 |
| -250 μm | 0.9884 | 0.0592 |
| +250 μm | 1.0000 | 0.0631 |

**Table 4**
Determination of Sintering, Softening and Melting Temperatures

| Particle Size | Sintering Temperature (°C) | Softening Temperature (°C) | Melting Temperature (°C) |
|---|---|---|---|
| 1993 Coal Ash | | | |
| 93-NSCFA* | 1093 | 1183 | >1500 |
| 93-1, +250 μm | 1230 | 1288 | >1500 |
| 93-2, 150~250 μm | 1218 | 1285 | >1500 |
| 93-3, 106~150 μm | 1220 | 1281 | >1500 |
| 93-4, 75~106 μm | 1206 | 1270 | >1500 |
| 93-5, 53~75 μm | 1230 | 1280 | >1500 |
| 93-6, 45 ~53 μm | 1206 | 1258 | >1500 |
| 93-7, 32~45 μm | 1115 | 1208 | >1500 |
| 93-8, -32 μm | 1120 | 1193 | >1500 |
| 1993 Coal Ash | | | |
| 96-NSCFA* | 1085 | 1156 | 1440 |
| 96-1, +106 μm | 1117 | 1194 | >1500 |
| 96-2, 75~106 μm | 1180 | 1242 | >1500 |
| 96-3, 53~75 μm | 1168 | 1220 | >1500 |
| 96-4, 48~53 μm | 1147 | 1203 | >1500 |
| 96-5, 38~48 μm | 1105 | 1175 | 1490 |
| 96-6, -38 μm | 1102 | 1160 | 1450 |

*NSCFA - No Sieving Coal Fly Ash

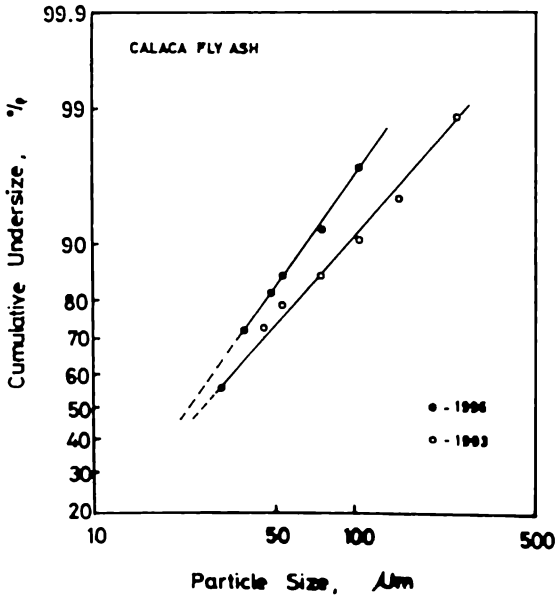


**Figure 1.** Distribution of Particle Size

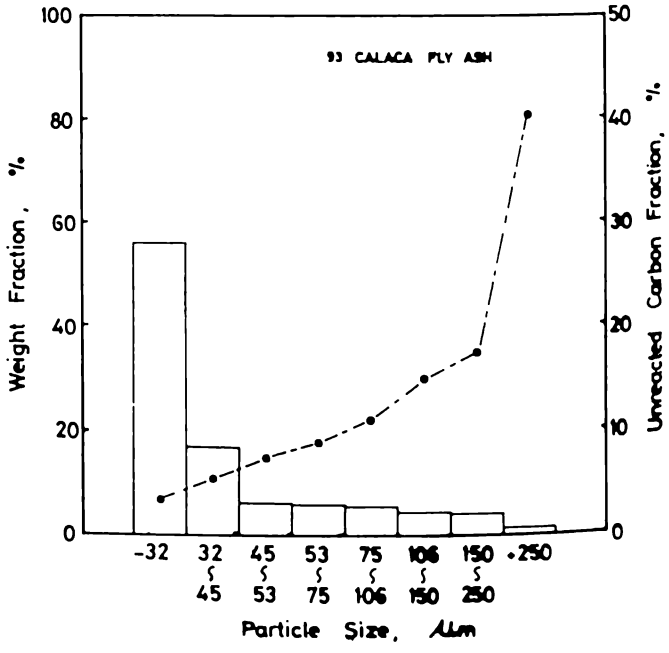


**Figure 2.** Weight Fraction vs. Unreacted Carbon for each Particle Size (1993 Coal Ash Samples)

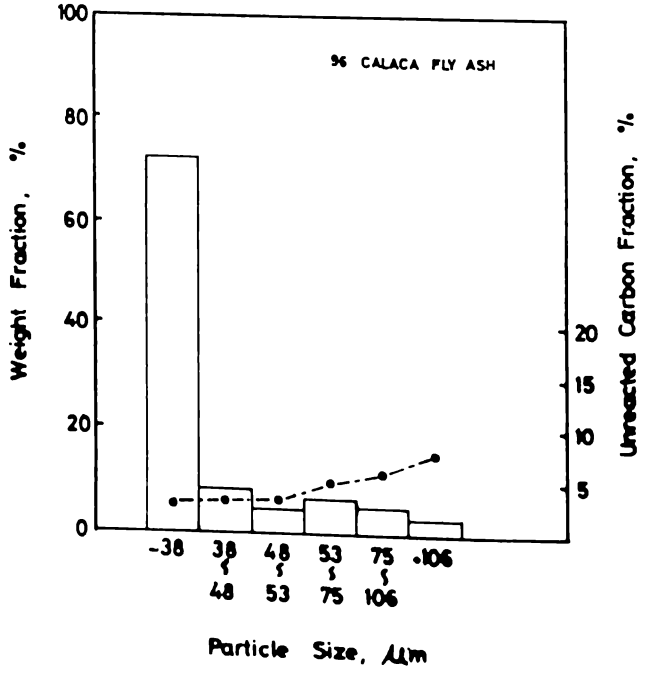


**Figure 3.** Weight Fraction vs. Unreacted Carbon for each Particle Size (1996 Coal Ash Samples)

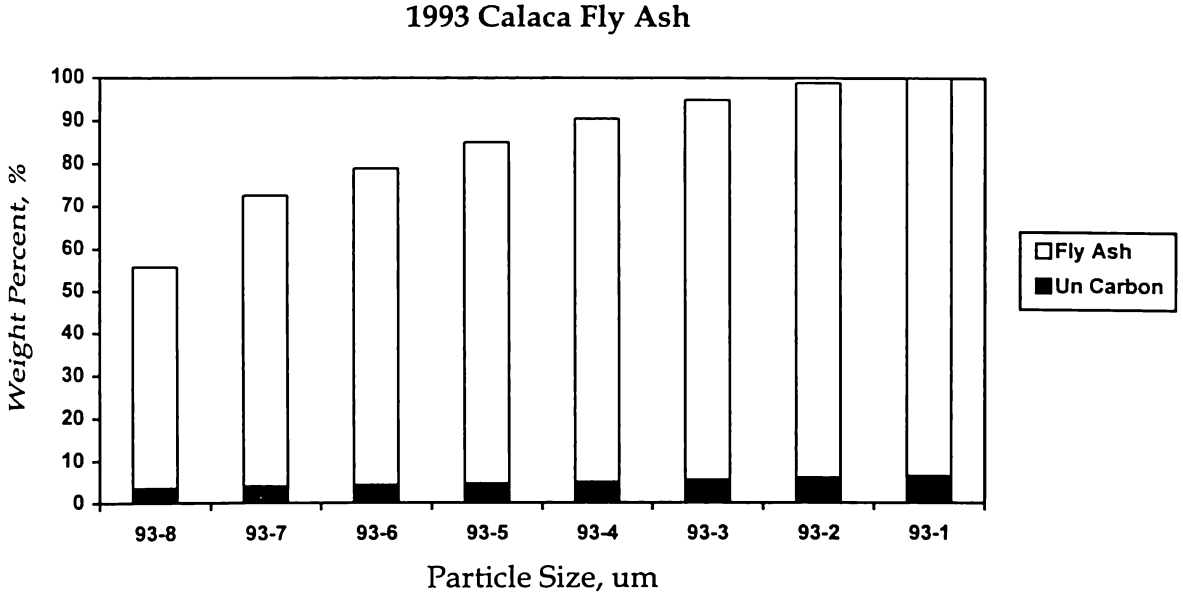


**Figure 4.** Cumulative Weight Fraction vs. Unreacted Carbon for each Particle Size (1993 Coal Ash Samples)

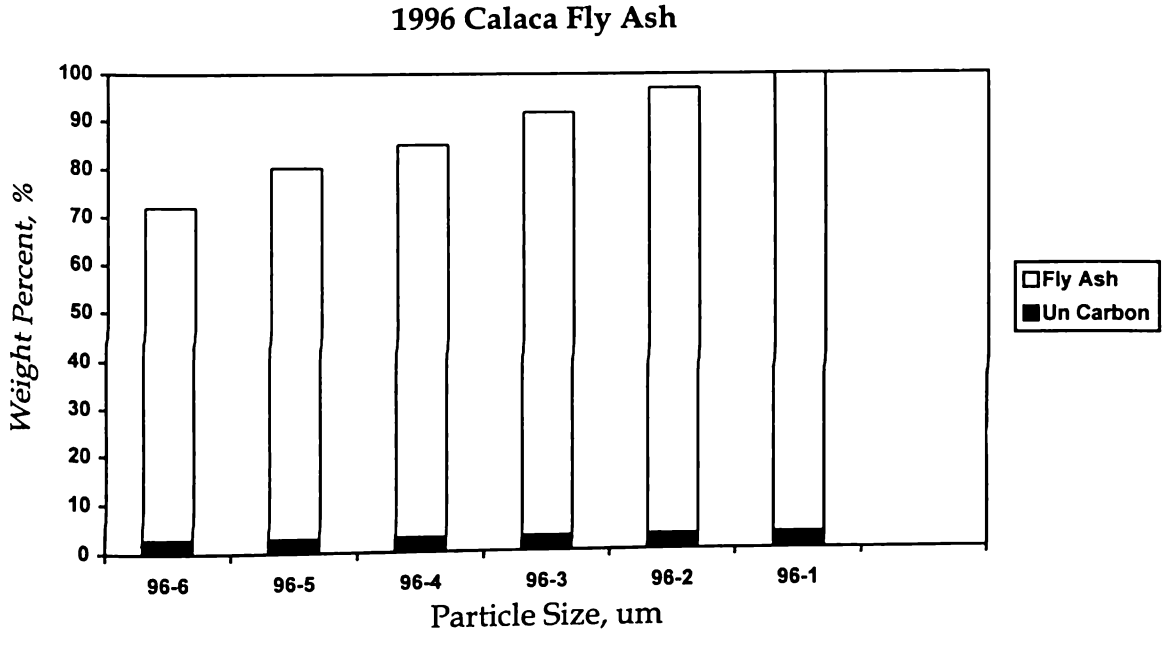


**Figure 5.** Cumulative Weight Fraction vs. Unreacted Carbon for each Particle Size (1996 Coal Ash Samples)

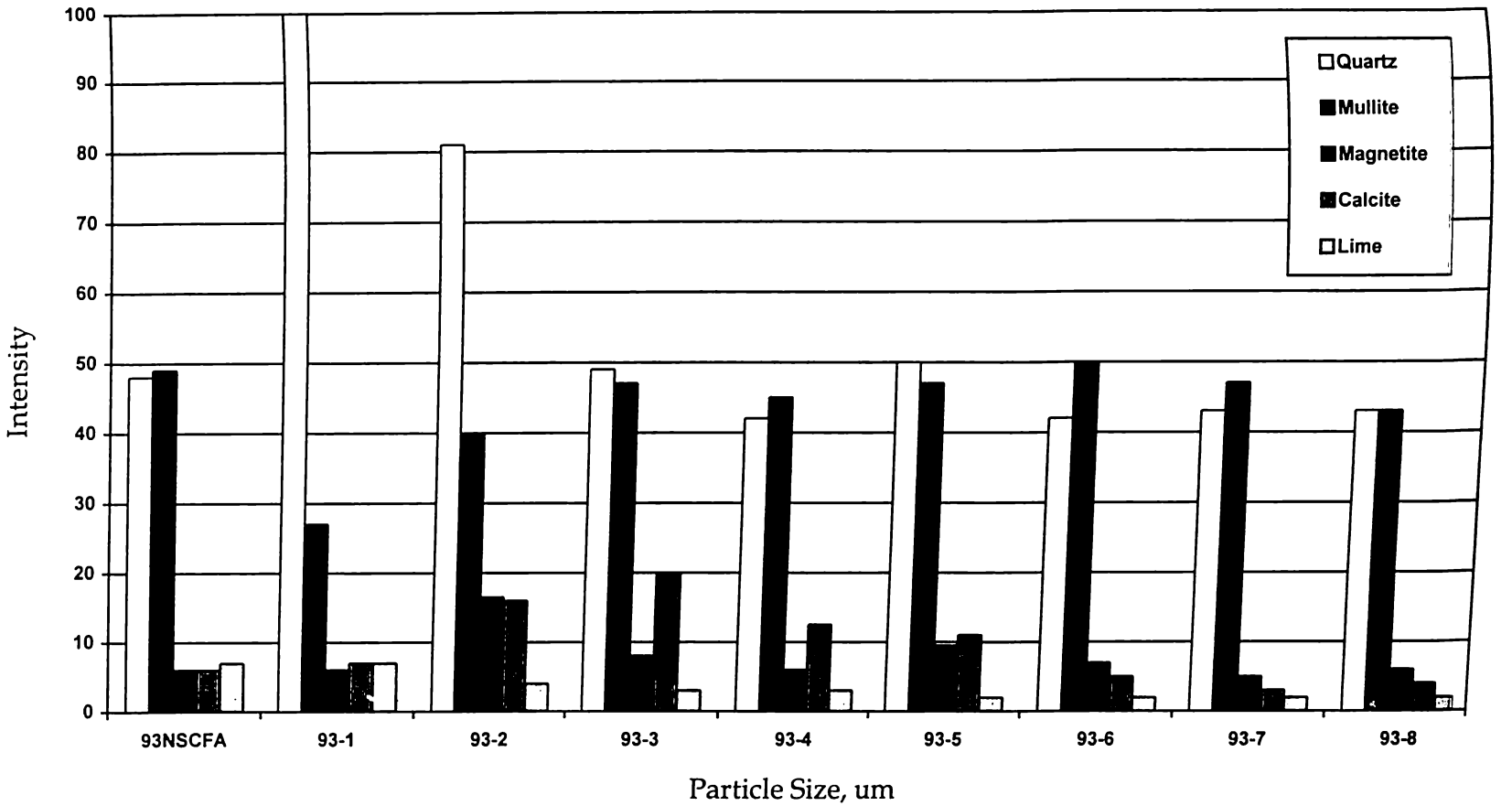


**Figure 6.** X-ray Diffraction Line Intensity of Major Elements for each Particle Size of the 1993 Coal Ash Samples

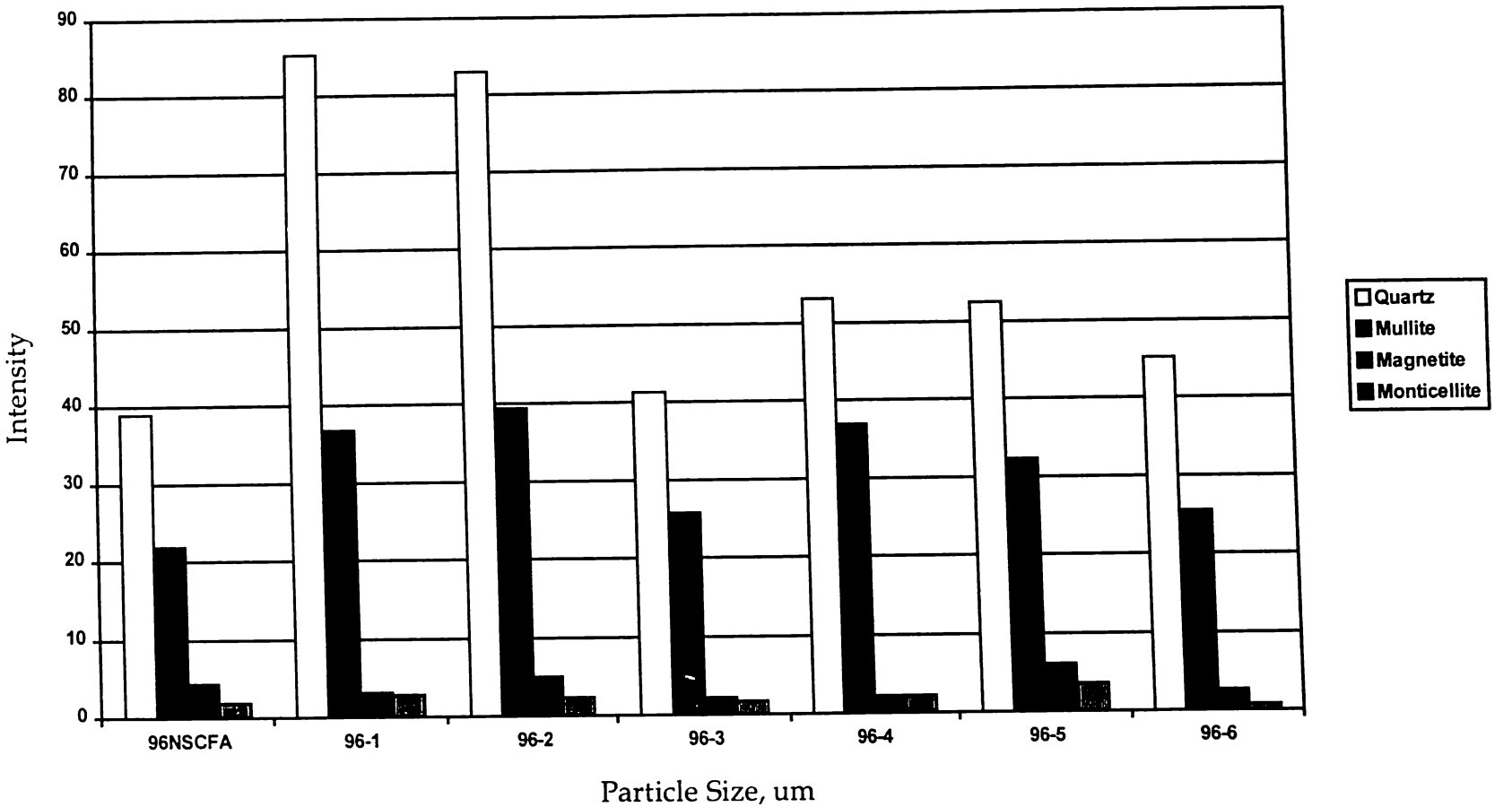


**Figure 7.** X-ray Diffraction Line Intensity of Major Elements for each *Particle Size of the 1996 Coal Ash Samples*

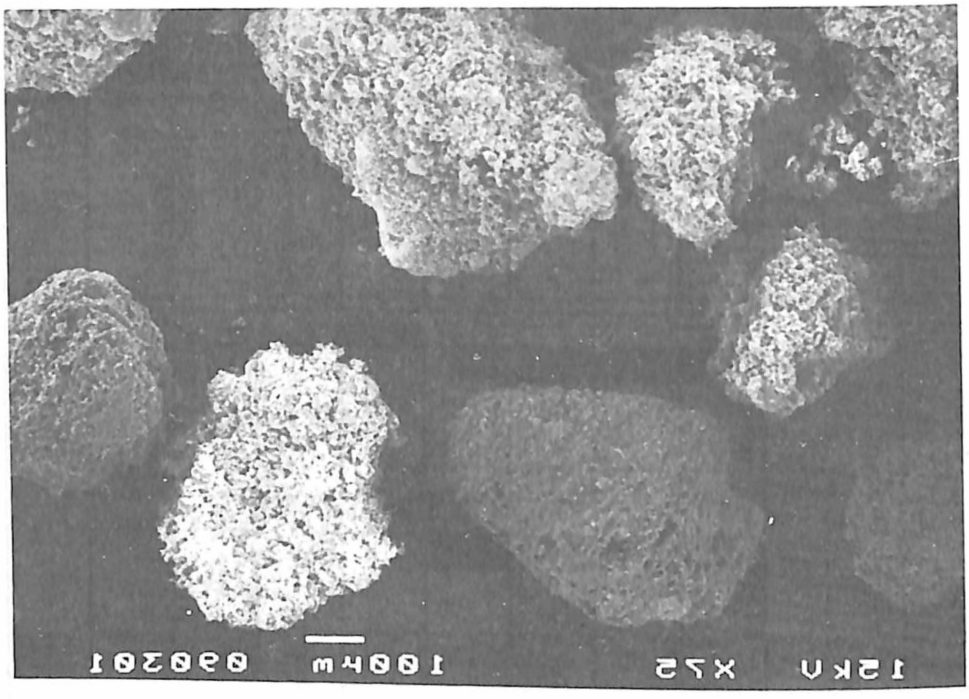

**Figure 8a.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: +250 $\mu$m)

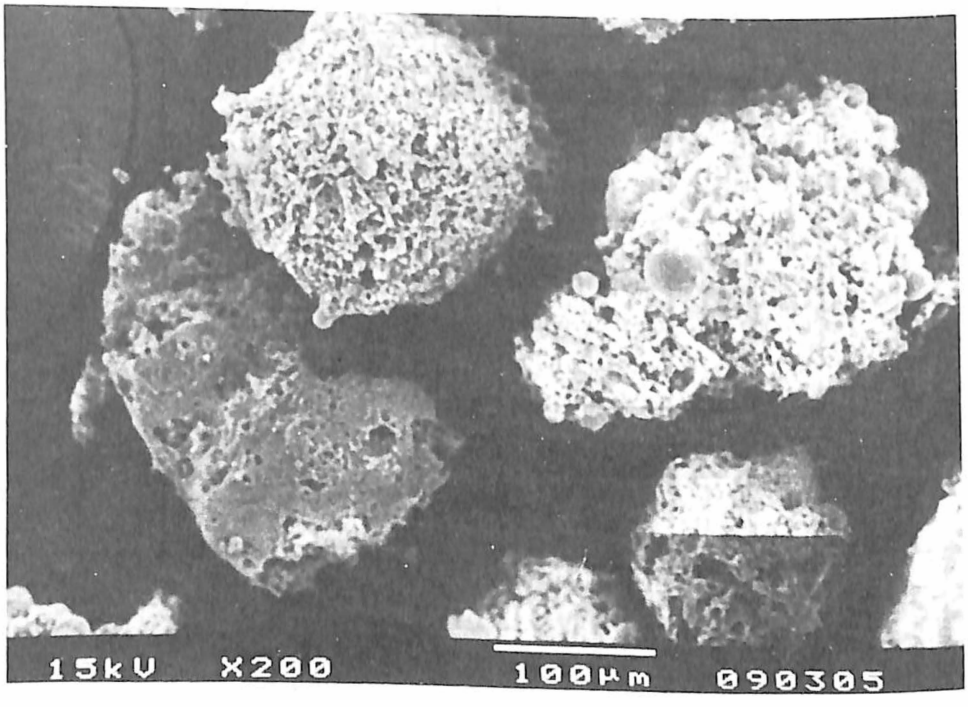


**Figure 8b.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: 150~250 $\mu$m)

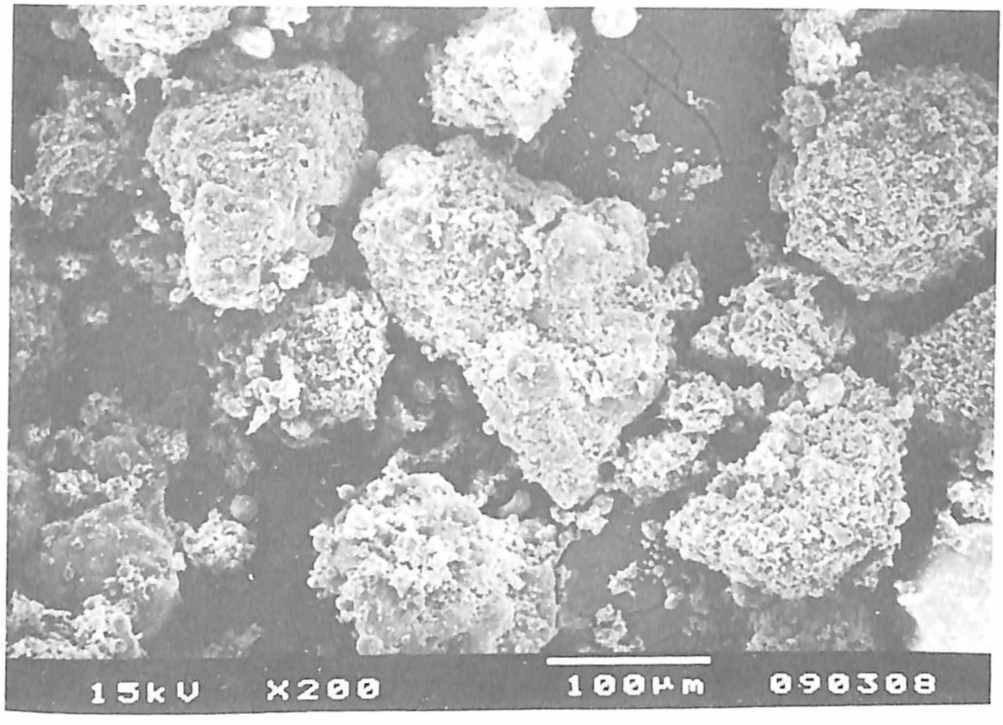


**Figure 8c.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: 106~150 $\mu$m)

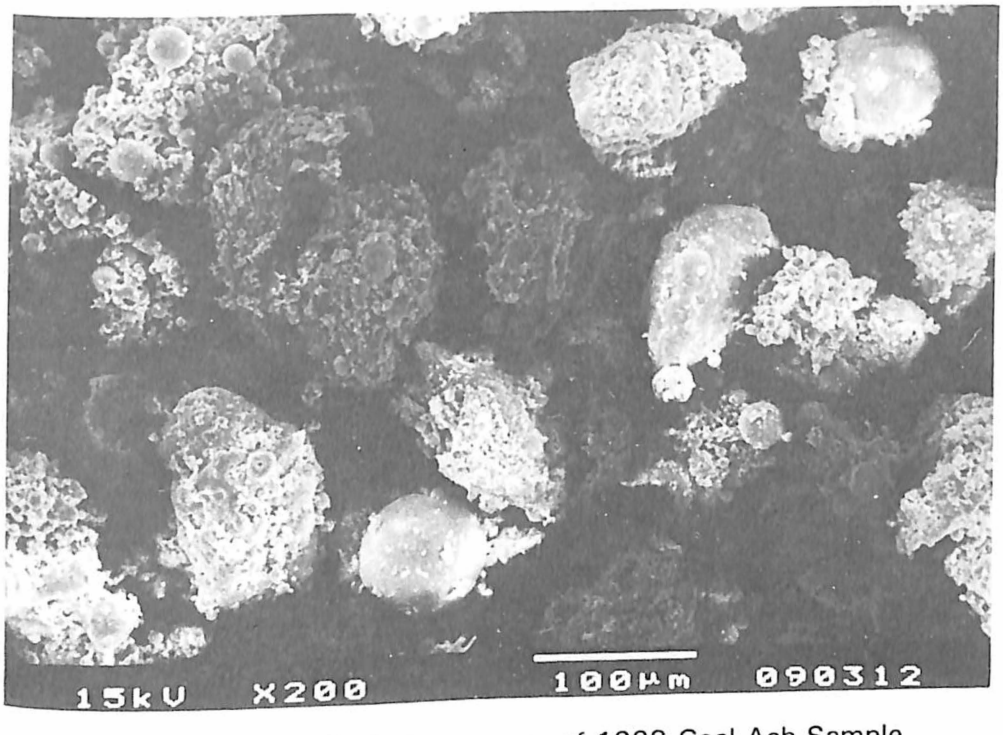


**Figure 8d.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: 75 ~106 $\mu$m)

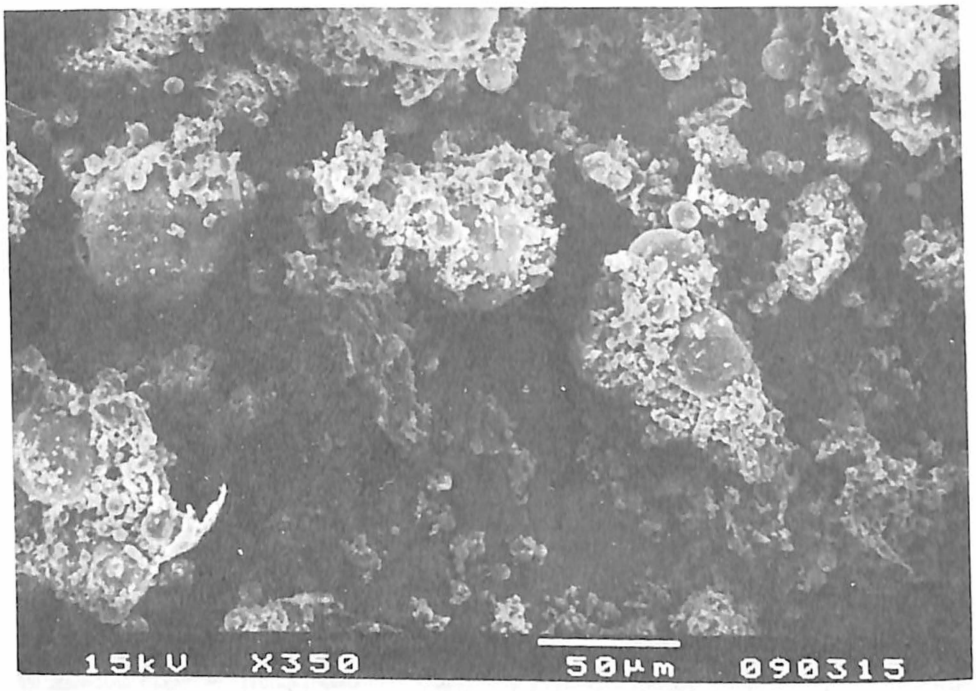


**Figure 8e.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: 53 ~75 $\mu$m)

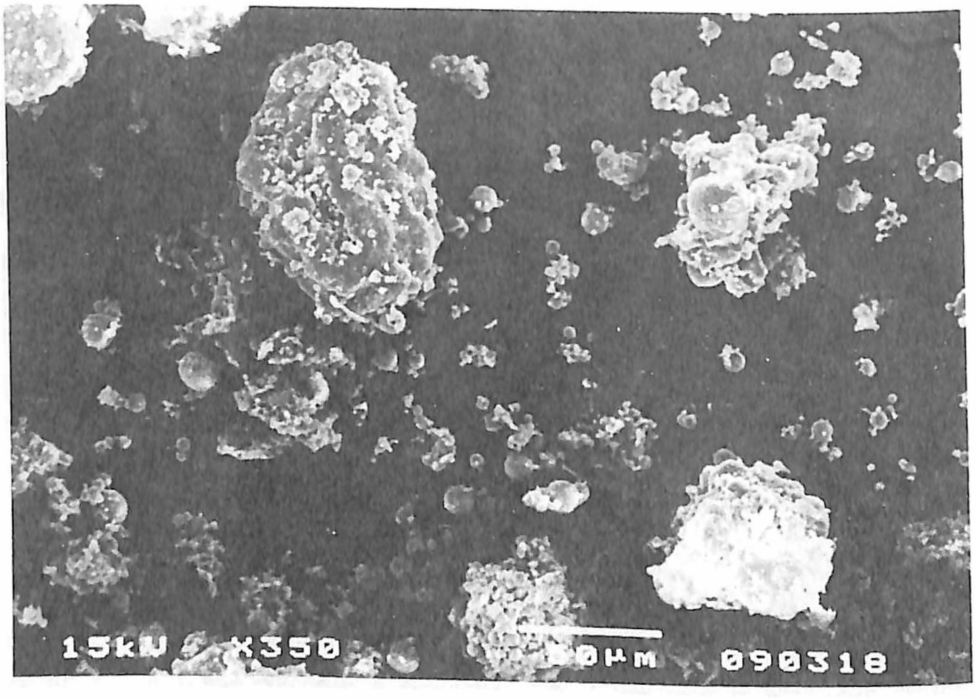


**Figure 8f.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: 45~ 53 $\mu$m)

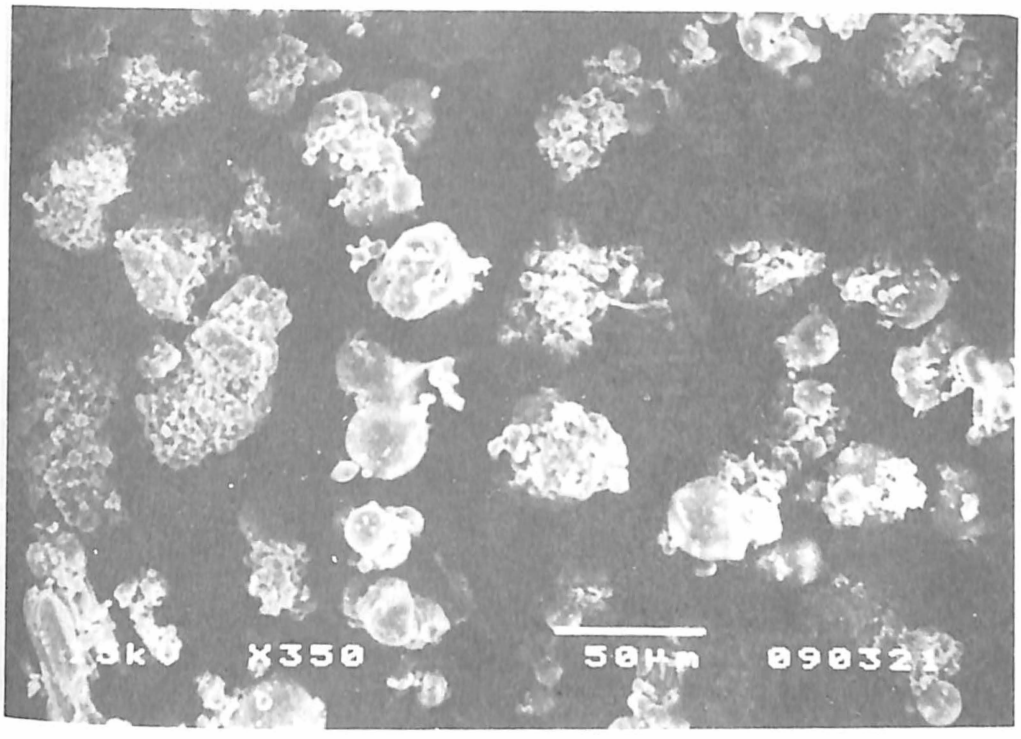


**Figure 8g.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: 32~45 $\mu$m)

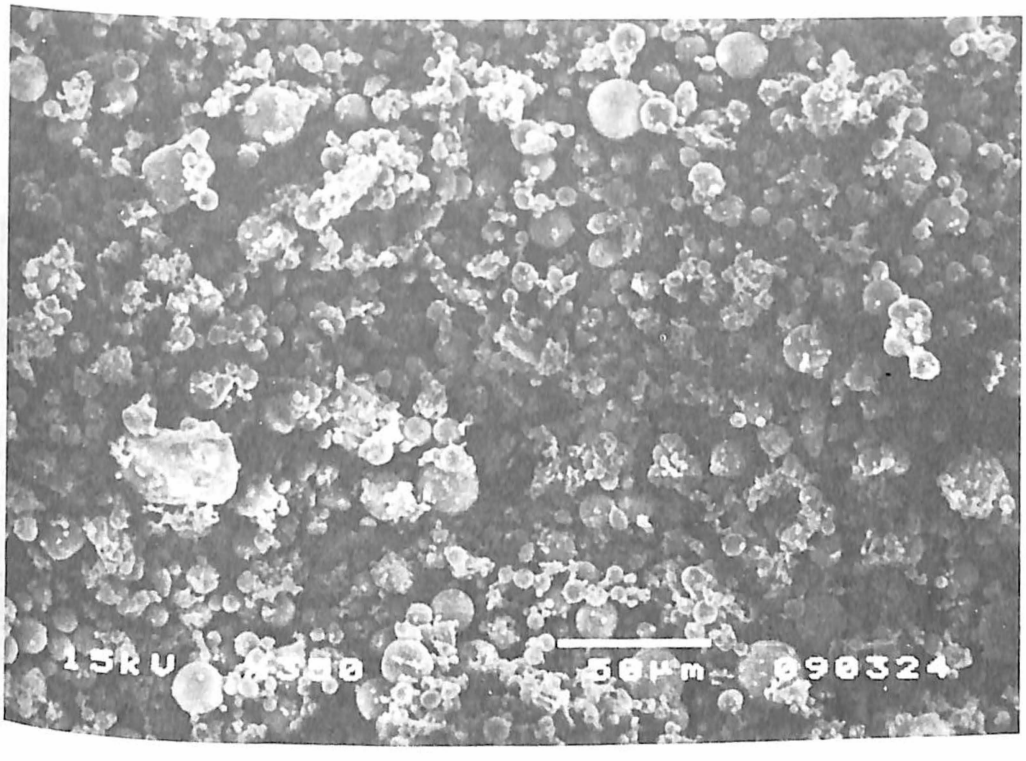


**Figure 8h.** Morphological Structure of 1993 Coal Ash Sample
(Particle Size: - 32 $\mu$m)

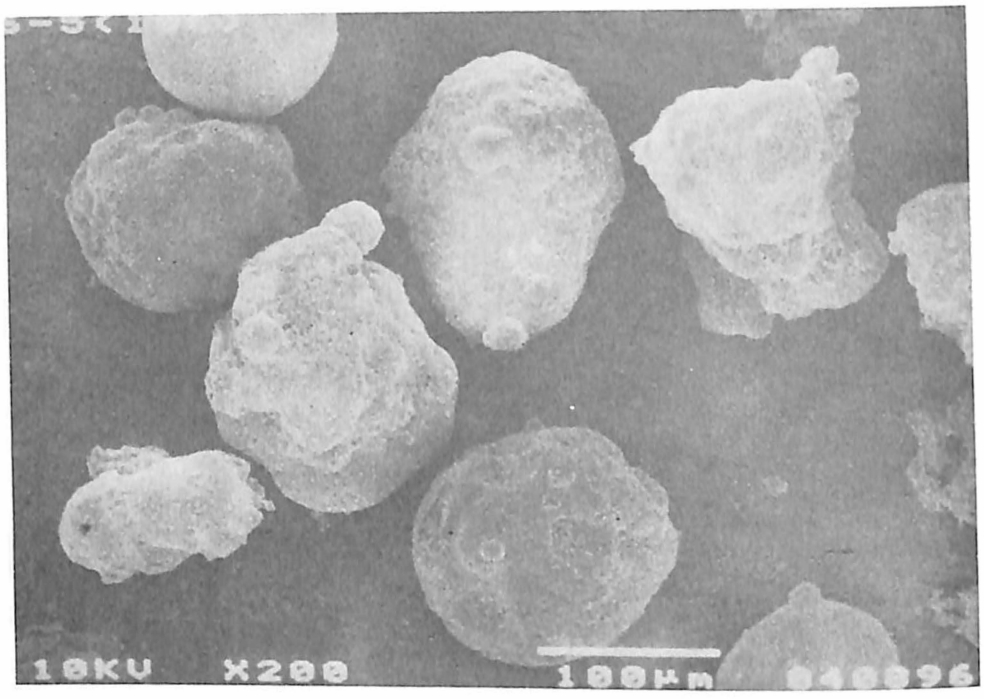


**Figure 9a.** Morphological Structure of 1996 Coal Ash Sample
(Particle Size: +106 $\mu$m)

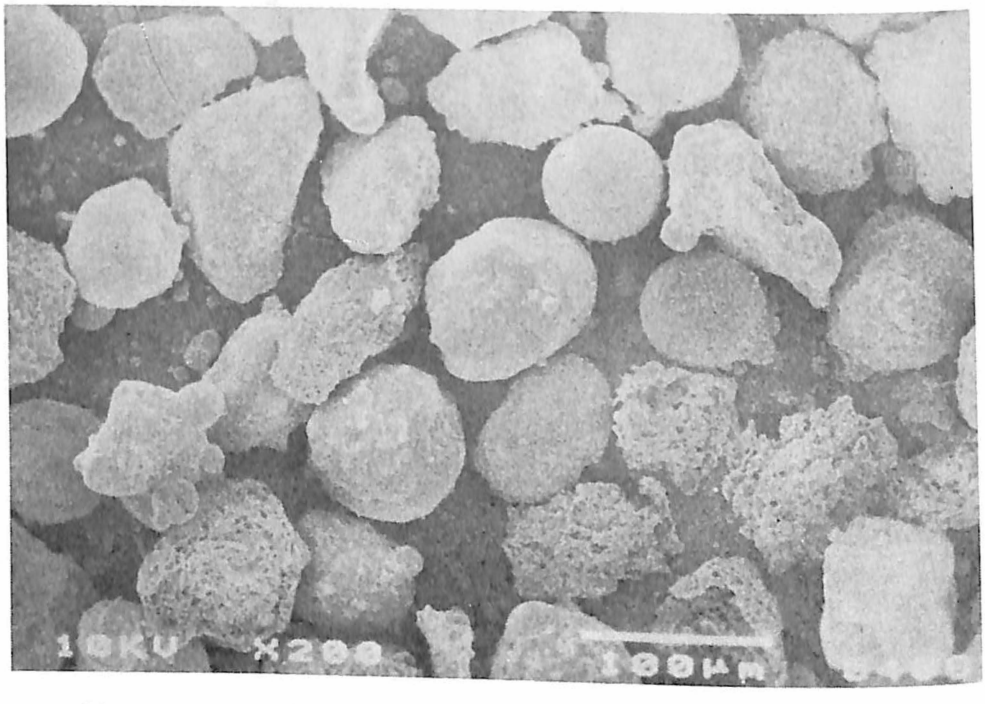


**Figure 9b.** Morphological Structure of 1996 Coal Ash Sample
(Particle Size: 75~106 $\mu$m)

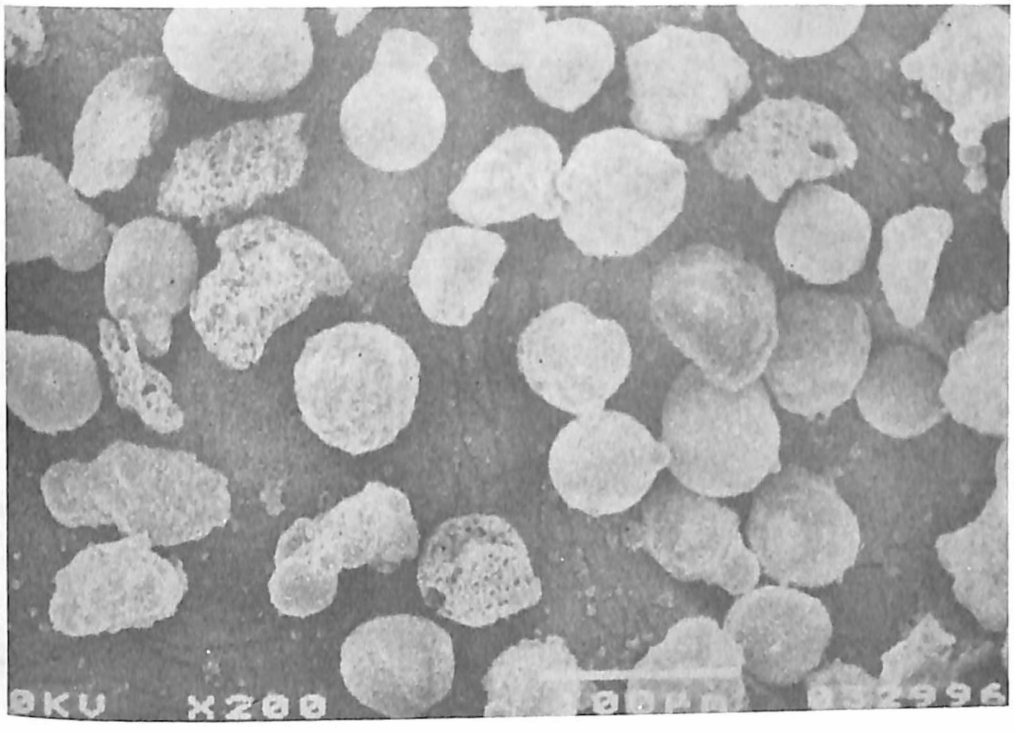


**Figure 9c.** Morphological Structure of 1996 Coal Ash Sample
(Particle Size: 53~75 $\mu$m)

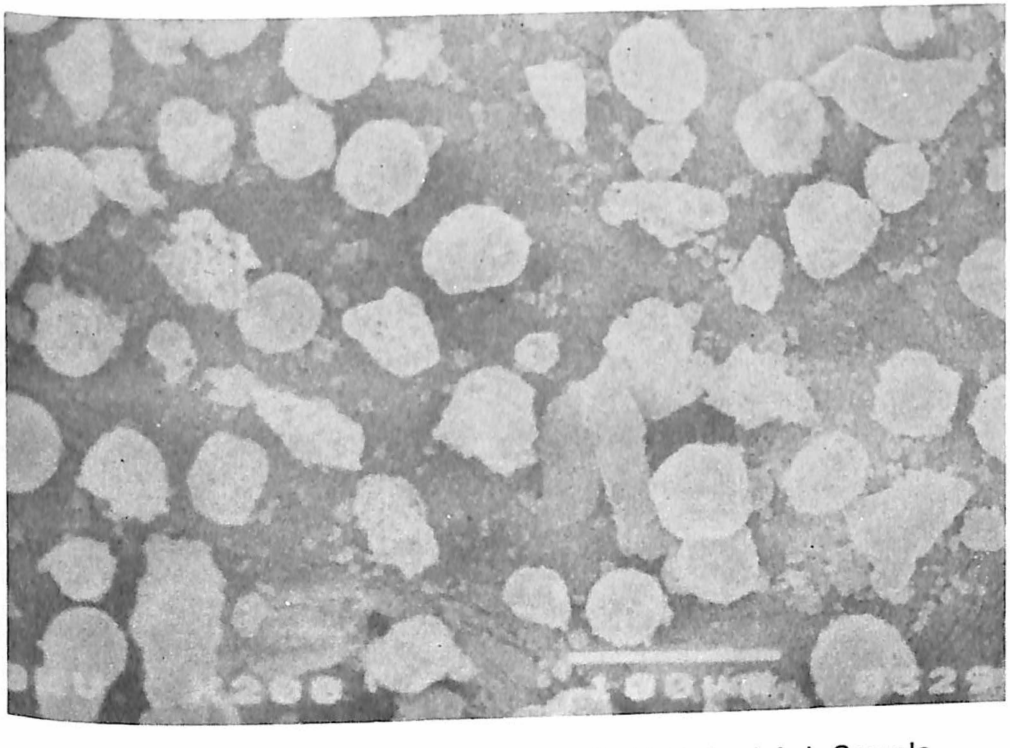

**Figure 9d.** Morphological Structure of 1996 Coal Ash Sample
(Particle Size: 48~53 $\mu$m)

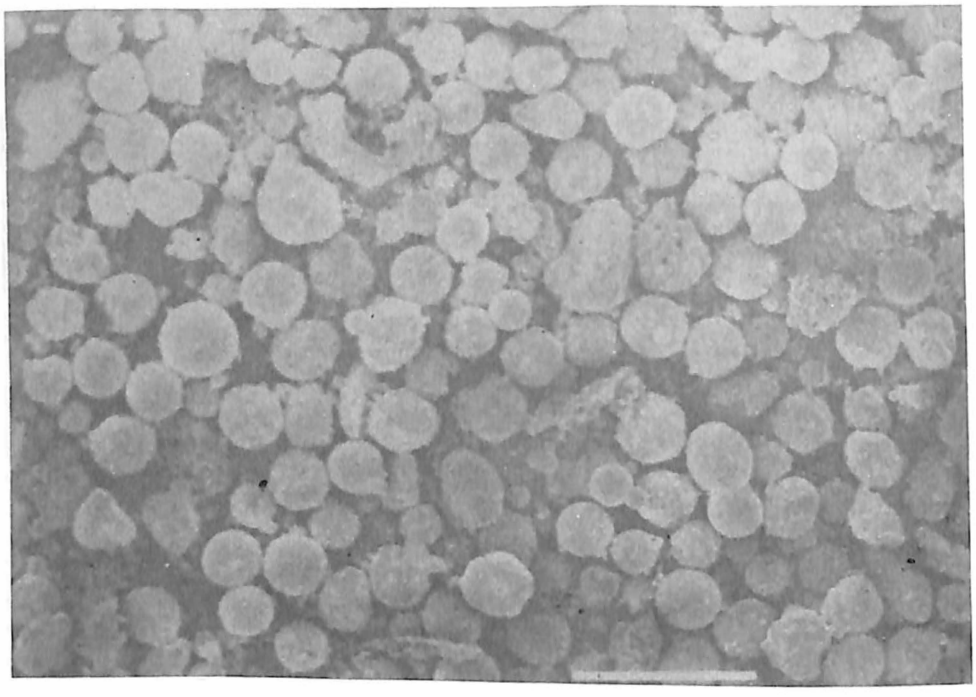

**Figure 9e.** Morphological Structure of 1996 Coal Ash Sample
(Particle Size: 38~48 $\mu$m)

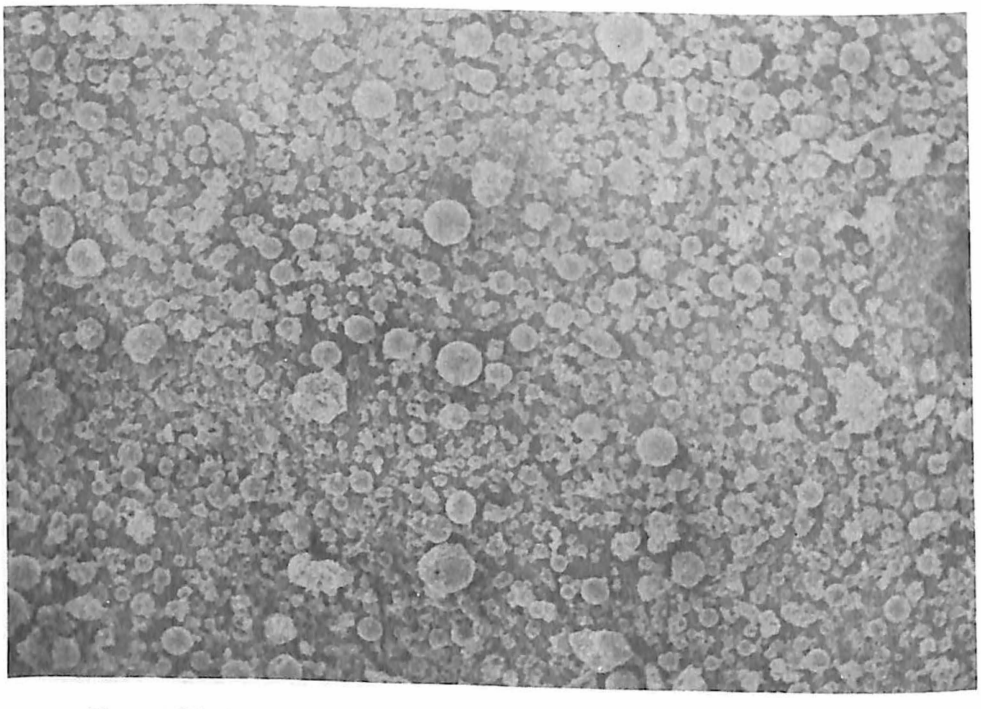

**Figure 9f.** Morphological Structure of 1996 Coal Ash Sample
(Particle Size: - 38 $\mu$m)

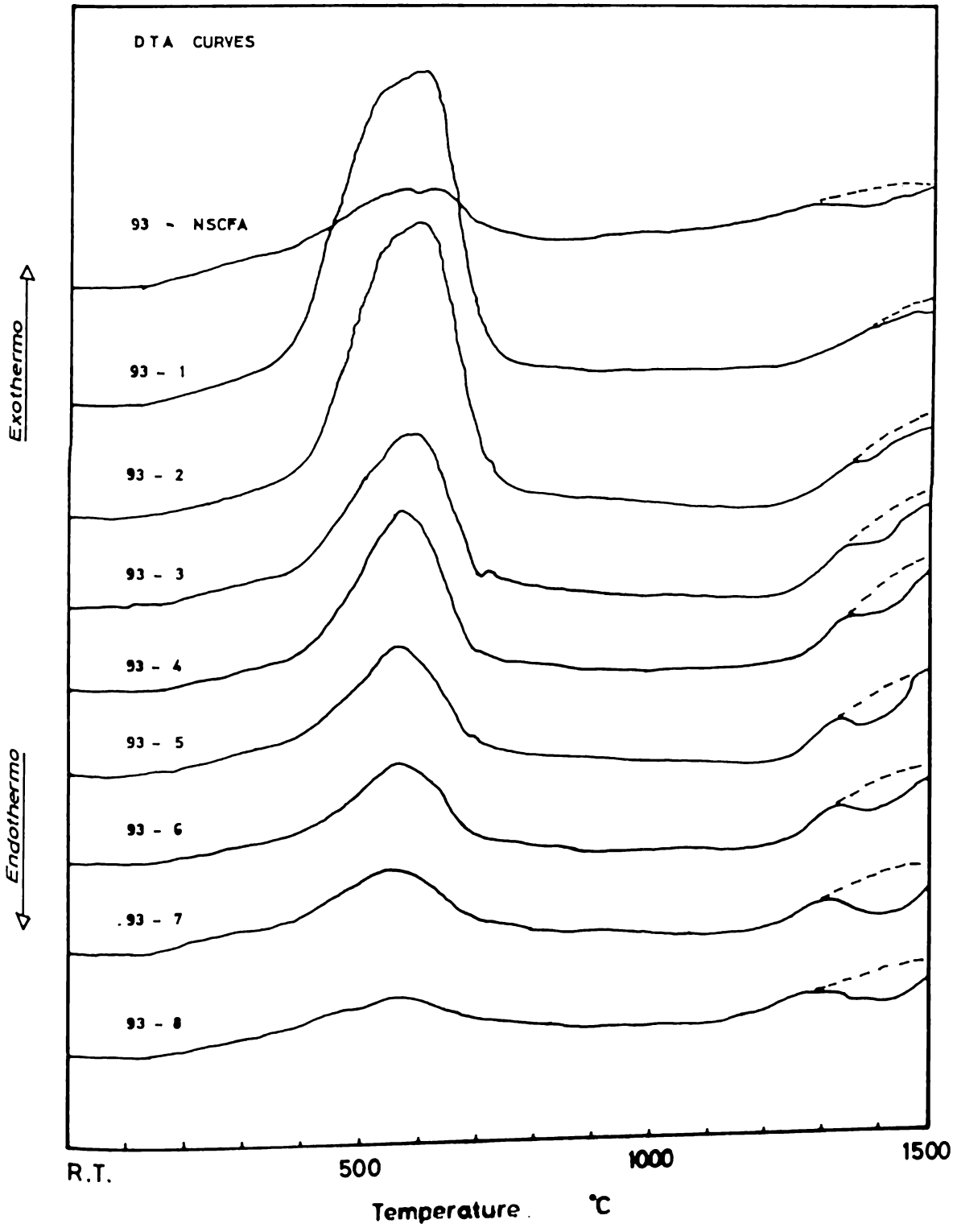


**Figure 10.** Thermal Analysis of each Particle Size of the 1993 Coal Ash Samples

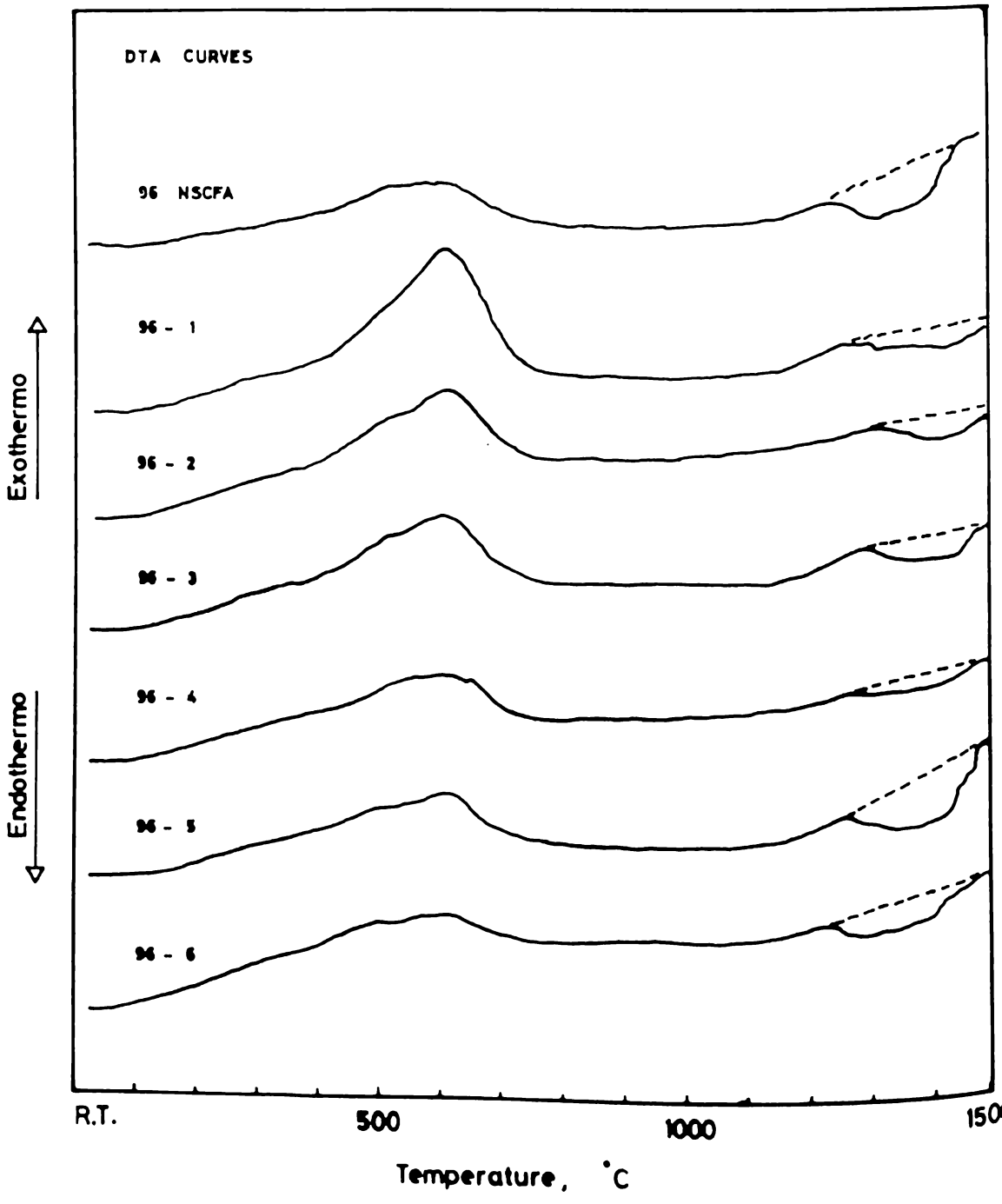


**Figure 11.** Thermal Analysis of each Particle Size of the 1996 Coal Ash Samples