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Studies on Coal Slurry Fuel (Part 4) Effects of Additive and Particle Size Distribution on Characteristics of Coal-Water Mixture (CWM)

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Six anionic polyelectrolytes (poly(styrenesulfonate) (PSS), three poly(isoprenesulfonate)s (PIPSs), a copolymer of sodium poly(methacrylate) and poly(methacrylate) ester (PMA), and a melamine-formaldehyde condensate sulfonate (MFCS)) were tested for additives for coal-water mixtures (CWMs), which were prepared either by dry grinding method or by wet grinding method, and evaluated by their viscosity and storage stability. A poly(isoprenesulfonate) PIPS-1 was found as effective as PSS, which is now most commonly used in practical CWM processes. A new parameter "liquid-absorption" is proposed. The parameter is defined as the minimum quantity of liquid (water:ethylene glycol 4:6) necessary for a certain amount of powdery coal to form a putty-like lump when the liquid is titrated on the powdery coal. Liquid-absorption reveals the quantity of the liquid that is tightly retained on the surface of coal particles and locked in the apertures of the particles. Various powdery coals, for which their mean particle sizes were adjusted to be about the same while their size distributions remained different, were prepared. For CWMs prepared from powdery coals, when the size of coal particles was more distributed, the liquid-absorption was reduced and the CWM viscosity was decreased. By use of powdery calcium carbonates, it was found that liquid-absorption for the powders was influenced both by the magnitude of the apertures of the particles and by the specific surface area.

1. Introduction

Coal-water mixture (CWM), which is composed of powdery coal and water, is an attractive utilization of coal and one of the most promising alternatives for heavy oil^{1)~4)}. In Japan the production of CWM has already become practical. Its commercial production (500,000 t/yr) has been realized by the Japan COM Co., Ltd. at Onahama, Fukushima. The CWM produced is transported through a pipeline to the Nakoso Power Station of Joban Joint Power Co., Ltd. It is planned that its production will be expanded to several million t/yr in a few years.

The feature characteristics of CWM are fluidity and storage stability without sedimentation. For practical use, CWM needs to contain 65-70 wt% of coal and its viscosity should be less than 1.0 Pa·s. In order to attain such characteristics, the following factors are important: (i) coal properties, (ii) particle size and its distribution, and (iii) additives which work as solid dispersants^{2),3)}.

Many studies have been made recently on each of the factors^{5)~20)}. In these studies naphthalenesulfonate-formaldehyde condensates (NSFs) and poly(styrenesulfonate)s have almost exclusively been used as CWM additives, and as a result, only a few studies on other additives have been reported. However, one of the major problems in practical use of CWMs is the relatively high costs of poly(styrenesulfonate)s which are superior to NSFs as additives and thus are most commonly utilized now.

Several reports have described the effect of size distribution of coal particles on the CWM properties^{5),6),15)}. However, in these studies, the distinction between effects of size and distribution is not clear. CWMs from various powdery coals of different size distributions and different mean particle sizes have been prepared.

Thus, factors (ii) particle size and its distribution and (iii) additives should deserve further exploration. In this study we have prepared various CWMs by use of a poly(styrenesulfonate) (PSS) as well as other less costly anionic polyelectrolytes, such as poly(isoprenesulfonate)s (PIPSs), a copolymer of sodium poly(methacrylate) and

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poly(methacrylate) ester (PMA), and a melamine-formaldehyde condensate sulfonate (MFCS). Moreover, we prepared CWMs from various powdery coals for which their size distributions were different but their mean particle sizes are adjusted to be about the same. We will further discuss such CWM properties as viscosity and storage stability in terms of the kinds of additive and the particle size distribution involved.

2. Experimental

2.1. Materials

Three coals listed in **Table 1** were tested. Six polyelectrolytes which included PSS, three PIPs (PIPS-1, PIPS-2, and PIPS-3), PMA, and MFCS were used as CWM additives. Polyelectrolytes, PSS, PIPs, PMA, and MFCS were donated by Lion Corporation, Japan Synthetic Rubber Co., Ltd., Nippon Shokubai Co., Ltd., and Showa Denko Co., Ltd. Structures and molecular weights of these polyelectrolytes are indicated in **Fig. 1**.

Three powdery calcium carbonates, NS-3000, SS-80, and NN-200, which had mean particle sizes of 2.0, 5.0, and 19 μm , respectively, were obtained from Nitto Funka Co., Ltd.

2.2. Preparation of Highly Loaded CWM

Coal of *ca.* 3.0 mm in diameter was pulverized in a ball mill. Particle size of the powdery coals used in the CWM was adjusted by blending coals of less than 74 μm and 74-125 μm in the weight ratio 80:20. The powdery coal thus prepared was slowly added to the aqueous solution containing the additive using a mixer with four blades (1000 rpm; 20 min; Tokushu Kikakogyo HV-M). The mixture was further stirred (4000 rpm) for 10 min. The composition of the CWM was coal (52-71 wt%), water (47.5-28.5 wt%), and the additive (0.5 wt%). The dry grinding method³⁾ was used.

In the wet grinding method^{3),7)}, pulverization was carried out in the presence of an additive and water until 80-83% of coal passed through a 74 μm mesh screen to produce a slurry. An appropriate amount of water was then added and the resulting slurry was stirred with a mixer (4000 rpm) for 10 min to prepare the CWM whose composition

was coal (63-68 wt%), water (36.5-31.5 wt%), and additive (0.5 wt%).

2.3. Apparent Viscosity of CWM

Apparent viscosity of CWM was measured using a rheometer with a coaxial rotating cylinder (Haake Rotovisco RV2). The shear history was as follows: shear rate $0 \rightarrow 150 \text{ s}^{-1}$ (3 min), 150 s^{-1} (3 min), $150 \rightarrow 0 \text{ s}^{-1}$ (3 min). The apparent viscosity was obtained from shear stress at 100 s^{-1} on the downward curve.

2.4. Storage Stability

In order to evaluate the storage stability of CWM, the "slurry discharge test" was performed according to the method given in the literature⁷⁾. A 200 g of CWM, which had viscosity of 1.0 Pa·s, was stored in a 350 ml polystyrene bottle for a given period. The bottle was inclined for 10 min at an angle of 60° above a 1 mm mesh screen placed on a beaker. A portion of the slurry passed through the screen was defined as the "discharged slurry".

	$\overline{M}_w$	$\overline{M}_w/\overline{M}_n$
PSS	16,000	3.6
PIPS	20,000 33,000 83,000	1.8 2.7 2.8	(PIPS-1) (PIPS-2) (PIPS-3)
PMA	20,000	2.0
MFCS	40,000 -50,000	a)	

a) unknown.

Fig. 1 Structures of Polyelectrolytes Used for CWM Additives

Table 1 Analysis of Coals

Coal ^{a)}	Proximate [%] ^{b)}					Ultimate [%]					
	IM	Ash	VM	FC	FR	C	H	N	S	O	O/C
A	4.2	9.1	31.7	55.0	1.74	85.1	5.2	1.8	0.6	7.3	0.086
B	3.3	10.3	33.5	56.3	1.68	81.1	5.1	1.2	0.3	12.3	0.152
C	6.0	13.8	40.9	45.4	1.11	76.3	5.8	1.3	0.6	16.0	0.210

a) A and C: Australia; B: China.

b) IM: Inherent moisture; VM: Volatile matter; FC: Fixed carbon; FR: Fuel ratio.

Table 2 Mixing Ratios, Mean Particle Sizes, and Particle Size Distributions for Powdery Coals M1-M4

Powdery coal	Mixing ratio of P1-P5 [%] ^{a)}					Mean particle size [μm]	α [$^{\circ}$] ^{b)}
	P1	P2	P3	P4	P5		
M1	2.3	4.7	11.6	23.3	58.1	19.8	34.4
M2	0	0	21.7	43.5	34.8	20.3	40.1
M3	0	5.9	0	94.1	0	20.4	44.8
M4	0	0	11.1	88.9	0	20.1	47.6

a) Mean particle sizes of powdery coals P1-P5 were 130, 92.4, 53.9, 15.9, and 4.5 μm , respectively.

b) Distribution constant (slope of Rosin-Rammler plot).

Table 3 Mixing Ratios, Mean Particle Sizes, and Particle Size Distributions for Powdery Calcium Carbonates C1-C7

Powdery CaCO_3	Mixing ratio [%] ^{a)}			Mean particle size [μm]	α [$^{\circ}$] ^{b)}
	NN200	SS80	NS3000		
C1	46.5	0	53.5	10.3	36.1
C2	39.4	30.3	30.3	9.9	39.2
C3	36.3	45.5	18.2	10.2	40.8
C4	34.9	51.2	13.9	9.8	43.2
C5	31.2	59.4	9.4	9.8	46.9
C6	30.3	63.6	6.1	10.0	49.0
C7	31.2	68.8	0	10.0	51.1

a) Mean particle sizes of commercially available powdery calcium carbonates NN200, SS80, and NS3000 were 19, 5.0, and 2.0 μm , respectively.

b) Distribution constant (slope of Rosin-Rammler plot).

2. 5. Preparation of Powdery Coals of Various Particle Size Distributions

Powdery coals of various particle size distributions, for which the mean particle sizes were adjusted to be about the same, were prepared. Coal of *ca.* 3.0 mm in diameter was pulverized in a ball mill. The resulting powdery coal was sieved through several wire mesh screens and then fractionated into five particle size groups P1-P5 that included 150-105 μm (P1), 105-74 μm (P2), 74-44 μm (P3), and under 44 μm . The fraction under 44 μm was further fractionated by natural sedimentation into two groups (P4: sedimented; P5: not sedimented). Mean particle sizes of the five powdery coal groups were measured using a centrifugal automatic particle analyzer (Horiba CAPA-500) with ethylene glycol as a dispersing medium. Mean particle sizes of P1-P5 were 130, 92.4, 53.9, 15.9, and 4.5 μm . Groups P1-P5 were mixed in ratios indicated in Table 2 to prepare four powdery coal groups M1-M4 for which the mean particle sizes were adjusted to be about the same (*ca.* 20 μm) while the particle size distributions remained different. Mean particle sizes and size distributions of the powdery coals were measured using the particle analyzer.

2. 6. Liquid-absorption

“Liquid-absorption” was measured in a similar manner which was used for “oil-absorption”²¹⁾ in coal-oil mixture (COM). A weighed amount of powder (sample *ca.* 5.0 g) was placed on a Petri dish. The powder was mixed by titrating a

mixture of water and ethylene glycol (4:6) using a spatula until a putty-like lump of 1.0 $\times$ 1.0 $\times$ 3.0 cm was formed. The amount of the liquid mixture titrated to the end point was converted to that per 100 g of sample, and it was defined as “liquid-absorption.”

2. 7. Preparation of Powdery Calcium Carbonate of Various Particle Size Distributions

Three commercially available powdery calcium carbonate powders of different mean particle sizes were mixed in the ratios indicated in Table 3 to prepare seven powdery calcium carbonate groups C1-C7 for which the mean particle sizes were adjusted to be about the same (*ca.* 10 μm) while the particle size distributions remained different. Specific surface areas of C1-C7 were measured using a Blaine air-permeation instrument (Kyushu Maruto; JIS R 5201-64) and their liquid-absorptions were measured in a similar manner.

3. Results and Discussion

3. 1. Achieved Coal Concentrations of CWMs Using Various Additives

Highly loaded CWMs were prepared with various additives such as PSS, PIPS-1, PIPS-2, and PIPS-3 by dry grinding method. Apparent viscosities of these CWMs were measured by a rheometer with a coaxial rotating cylinder. For coals A, B, and C, plots of apparent viscosities against coal concentrations in the CWMs are presented in Fig. 2. The CWM viscosity for all

Table 4 Achieved Coal Concentration (ACC)

Additive	ACC [wt%]		
	Coal A	Coal B	Coal C
PSS ^{a)}	65.7	70.5	55.2
PIPS-1 ^{a)}	66.4	68.5	56.8
PIPS-2 ^{a)}	63.8	66.6	56.3
PIPS-3 ^{a)}	62.7	65.6	54.0
PSS ^{b)}	67.5		
PIPS-1 ^{b)}	67.4		
PMA ^{b)}	66.2		
MFCS ^{b)}	64.4		

a) Dry grinding method.

b) Wet grinding method.

CWMs were prepared from coals A, B, and C by dry grinding method.

Fig. 2 Plot of Apparent Viscosity against Coal Concentration

coals drastically increased as the coal concentration increased. The “achieved coal concentration (ACC)” is defined as the coal concentration of CWM at which its viscosity becomes 1.0 Pa·s. The ACC values for coals A, B, and C are recorded in Table 4.

For all four additives, ACC values decreased in the order: coal B>A>C. It is known that ACC values are dependent on lipophilicity-hydrophilicity factors for coals such as fuel ratio and inherent

CWMs were prepared from coal A by wet grinding method.

Fig. 3 Plot of Apparent Viscosity against Coal Concentration

moisture^{20,19)}. Actually in the present case, ACC values decreased as the inherent moisture increased.

For the three PIPs, the ACC values decreased as their molecular weights increased. Ukigai *et al.* reported also that for PSS which had molecular weight greater than 1.0×10^4 , the viscosity of CWM increased as the molecular weight of PSS increased¹⁸⁾. They described that increasing viscosity of the medium due to nonadsorbed PSSs and due to the aggregate formation by bridging of the adsorbed PSSs across the coal particles might further be enhanced as the molecular weight of PSS increased. For CWMs with PIPs, these phenomena might have also taken place, thus PIPS-1, which has the lowest molecular weight, was superior to higher molecular weight PIPs. Consequently, it was shown that PIPS-1 worked as well as PSS as a CWM additive.

By using the wet grinding method, CWMs were also prepared from coal A. The additives included PSS, PIPS-1, PMA, and MFCS. Recently the wet grinding method has become more popular than the dry grinding method in practical CWM processes³⁾. Plots of apparent viscosities against coal concentrations in CWMs are presented in Fig. 3, and the ACC values are also recorded in Table 4 whose values decrease in the order: PSS>PIPS-1>PMA>MFCS. It was found that PIPS-1 was as effective as PSS also in the wet grinding method. Polyelectrolytes PMA and MFCS also worked as additives for highly loaded CWMs in which the coal concentration was greater than 64 wt%.

3. 2. Storage Stabilities of CWMs Using Various Additives

In order to evaluate the storage stability of

Table 5 Amount of Discharged Slurry (ADS)

Additive	ADS [wt%]					
	7-day storage			14-day storage		
	Coal A	Coal B	Coal C	Coal A	Coal B	Coal C
PSS ^{a)}	87.3	84.9	1.0	80.6	78.2	2.1
PIPS-1 ^{a)}	89.0	93.0	0	85.0	86.2	0
PIPS-2 ^{a)}	88.0	91.0	8.0	85.0	85.4	0
PIPS-3 ^{a)}	75.0	93.0	10.0	70.0	85.6	0
PSS ^{b)}	86.2			78.9		
PIPS-1 ^{b)}	84.2			75.2		
PMA ^{b)}	80.8			73.5		
MFCS ^{b)}	75.2			62.3		

a) Dry grinding method.

b) Wet grinding method.

CWMs were prepared from powdery coals M1-M4 by dry grinding method.

Fig. 4 Plot of Apparent Viscosity against Coal Concentration

CWMs, the slurry discharge test⁷⁾ was performed. The amount of discharged slurry (ADS) for CWMs from coals A, B, and C using the dry grinding method are recorded in **Table 5**. CWMs from coals A and B, which showed high ACC values, were well discharged after 7 days and 14 days of storage.

In **Table 5** are also recorded the ADS values for CWMs prepared using the wet grinding method. The amount of discharged slurry decreased, as the additive was varied, according to PSS > PIPS-1 > PMA > MFCS, which parallels the ACC value.

3. 3. Properties of CWMs from Powdery Coals M1-M4

So far the effect of particle size distribution on the CWM properties has been studied^{5), 6), 15)}. However, in the studies the effects of particle size distribution and mean particle size cannot be clearly distinguished. Thus, in this study CWMs were prepared from various powdery coals for which the mean particle sizes were adjusted to be about the same while the particle size distributions remained different.

Fig. 5 Plot of Achieved Coal Concentration (ACC) against Distribution Constant α for Powdery Coals M1-M4

Table 6 Achieved Coal Concentration (ACC) and Amount of Discharged Slurry (ADS) for CWMs from Powdery Coals M1-M4

Powdery coal	ACC [wt%]	ADS [wt%]	
		7-day storage	14-day storage
M1	66.8	91.6	89.2
M2	64.3	44.4	30.2
M3	59.8	16.7	11.1
M4	60.0	15.3	10.7

Powdery coal from coal A which had been pulverized was sieved with several size wire mesh screens. The resulting powdery coals which were fractionated into five particle size groups were mixed to make four powdery coal groups, M1-M4, for which the mean particle sizes were adjusted to be almost the same (*ca.* 20 μm) while the particle size distributions remained different (**Table 2**). Distribution constants α for the Rosin-Rammler plots²²⁾ for the powdery coal groups M1-M4 are also recorded in **Table 2**.

For powdery coals M1-M4, plots of apparent viscosity against coal concentration in CWMs are presented in **Fig. 4**. The ACC values obtained for

powdery coals M1-M4 are recorded in **Table 6**. **Figure 5** shows the plot of ACC value against distribution constant α . Powdery coals which had a wider range of distribution provided higher ACC values. The slurry discharge test⁷⁾ was performed for these powdery coals and the ADS values are also recorded in **Table 6**. When powdery coals of wider size distributions were used, higher ADS values were obtained. Consequently, by use of powdery coals M1-M4, it was clearly demonstrated that size distribution was important in terms of flow and storage properties of CWM.

3. 4. Liquid-absorption for Powdery Coals M1-M4

It is anticipated that decrease in CWM viscosity due to the increase in size distribution may be caused by enhanced packing of coal particles in the CWM, which results in decrease in the quantity of immobilized water fraction on the coal particles. When the quantity of immobilized water fraction decreases, that of water fraction which can contribute a flow of CWM should increase, which leads to decrease in viscosity. To probe the effect of particle packing on the viscosity, we have investigated the minimum quantity of liquid which is required for a certain amount of powdery coal to form a putty-like lump.

For the study of coal-oil mixtures (COMs), we have proposed a parameter "oil-absorption" which is defined as the minimum quantity of B-heavy oil (g) necessary for 100 g of powdery coal to form a putty-like lump²¹⁾. Oil-absorption²¹⁾ is considered to indicate the quantity of immobilized oil fraction in the COM that is tightly retained on the surface of coal particles and locked in the apertures of the particles^{21), 23), 24)}. Oil-absorption²¹⁾ has been found to correlate well with the viscosity of COM²⁴⁾.

In our study on CWMs, we used a mixture of water and ethylene glycol (4:6) instead of B-heavy oil in the oil-absorption²¹⁾. Powdery coals do not provide putty-like lumps with only water, while pure ethylene glycol is quite hygroscopic. Thus a mixture of water and ethylene glycol was utilized for the liquid-absorption experiments. A parameter "liquid-absorption" here is defined as the minimum quantity of a mixture of liquids necessary for 100 g of powdery coal to form a putty-like lump.

The plot of liquid-absorption for powdery coals M1-M4 against distribution constant α is shown in **Fig. 6**. As the size distribution is increased, packing of coal particles is enhanced, and thus the apertures in the coal particles is reduced. However, increase in the size distribution also

Fig. 6 Plot of Liquid-absorption against Distribution Constant α for Powdery Coals M1-M4

Fig. 7 Plots of Specific Surface Area and Liquid-absorption against Distribution Constant α for Powdery Calcium Carbonates, C1-C7

provides an increase in the specific surface area, which may enhance liquid-absorption. As shown in **Fig. 6**, powdery coals, which have a wide range of size distribution, provide lower liquid-absorption. It appears that the influence of the decrease in aperture on the liquid-absorption surpasses that of the increase in specific surface area.

3. 5. Liquid-absorption for Powdery Calcium Carbonates

Influences of the magnitude of aperture and specific surface area on liquid-absorption have been further investigated by use of powdery calcium carbonates, which have surface properties different from those of coals. Powdery calcium carbonates, C1-C7, for which their mean particle sizes are adjusted to be about the same while their particle size distributions remained different, have been prepared by mixing three commercially available calcium carbonates. Values of distribution constant α for the Rosin-Rammler plots²²⁾ for C1-C7 are recorded in **Table 3**. For C1-C7, their

liquid-absorption and specific surface area were measured, and **Fig. 7** shows the plots of specific surface area and liquid-absorption against distribution constant α . Powdery calcium carbonates, which have a wider range of size distributions, provide a wider range of specific surface area. It appears that as the range of size distribution becomes wider, the number of smaller particles increases, and the specific surface area is increased.

When the values of α of powdery calcium carbonates are between 52-41°, liquid-absorption for the powder decreases with increase in the value of α as is observed for powdery coals M1-M4 (**Fig. 6**). However, when the values of α are smaller than 41°, liquid-absorption increases. Thus, for powdery calcium carbonates C1-C7, it is proposed that liquid-absorption is influenced both by the magnitude of the aperture of the particles and by the specific surface area. The difference in the behaviors for powdery coals and powdery calcium carbonates may be ascribed to the difference in their mean particle sizes and/or in their surface properties.

3. 6. Conclusion

Highly loaded CWMs prepared by use of various polyelectrolytes have been tested from viscosity and storage stability. Polyelectrolyte PIPS-1 is as effective as PSS as an additive for CWMs. Polyelectrolytes PMA and MFCS also work as additives for highly loaded CWMs in which coal concentration is more than 64 wt%.

Powdery coals with a wider range of size distribution providing lower liquid-absorption give higher ACC values. It is indicated that the liquid-absorption proposed in this study can serve as a parameter to estimate the immobilized liquid fraction that is tightly retained on the surface of the particles and locked in their apertures. Such immobilized fraction in CWM cannot contribute to its flow; thus the viscosity of CWM is increased.

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要 旨

石炭スラリー燃料に関する研究（第4報）
石炭-水混合物（CWM）の性能に対する添加剤と粒度分布の効果

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石炭-水混合物（CWM）の添加剤として，6 種のアニオン性高分子電解質（ポリスチレンスルホン酸塩（PSS），3 種のポリイソプレンスルホン酸塩（PIPS），ポリメタクリル酸塩とポリメタクリル酸エステル共重合体（PMA），メラミン-ホルムアルデヒド縮合物のスルホン化物（MFCS））をテストした。CWM は乾式粉碎法および湿式粉碎法によって調製した。ポリイソプレンスルホン酸塩（PIPS-1）は，現在最も普通に CWM の実操業で使用されている添加剤ポリスチレンスルホン酸塩（PSS）と同じくらい効果的であった。新しいパラメー

ターである「吸液力」を定義した。吸液力とは液体（水：エチレングリコール 4:6）を 100 g の石炭粉末に滴下して，粉末が初めてパテ状の一つの塊となるまでに使用された液体の量と定義され，これは粒子表面および粒子間げきに保持される液体量を表す。平均粒径がほとんど同一で粒度分布が異なる石炭粉末を調製した。石炭粉末の粒度分布が広いほど，吸液力は小さく，また CWM の粘度は減少した。炭酸カルシウム粉末を用いる実験によって，吸液力は粒子間げきの度合いと比表面積の両方に影響されることがわかった。

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Keywords

CWM, Additive, Viscosity, Storage stability, Particle size distribution, Liquid-absorption