Ability of Surfactants to Form Highly Loaded Coal-Water Mixtures

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Coal tends to aggregate in water. In a coal-water mixture (CWM), the higher the coal content is, the more thickened the mixture is. To highly load a CWM with coal, therefore, it is necessary to use a dispersant. The authors synthesized new types of multibranched high molecular nonionic surfactants that did not contain any alkali metal which would be harmful in combustion. Those surfactants were added to CWM samples containing Tatung coal pulverized by a ball mill. As a result, it was found that they contributed greatly to improving the fluidity of CWM, as briefly described below.

(i) Alkylene-oxide-added multi-branched high molecular nonionic surfactants improve their ability to highly load a CWM with an increase in the molecular weight per active hydrogen atom. When the molecular weight is 3,000 to 6,000 or more, they can make a CWM containing 69% coal. (ii) Sodium salt of formalin naphthalenesulfonate condensate, a type of anionic surfactant, can increase the coal content only to a maximum of 65%. (iii) Multi-branched high molecular nonionic surfactants can make a stable CWM.

In recent years when anxiety over exhaustion of petroleum resources rose, we reviewed the use of coal resources whose reserves are estimated to be 10 times or more those of petroleum reserves. Particularly, a highly loaded CWM (coal-water mixture) is now studied eagerly in many countries, because it can alleviate the drawbacks of coal such as difficulty in transportation, tendency to generate dust and danger of spontaneous ignition.

However, this type of fuel has not yet been practically used. Only in Black Mesa, Arizona, a slurry containing as little as about 50% coal is transported. After transportation through the pipeline, this slurry is dehydrated before combustion. The troublesome process of dehydration and water treatment has prevented it from being widely applied. On the other hand, a CWM fuel under study is advantageous in that not only is direct combustion possible but also transportation and storage expenses can be saved.

In studies of CWM, it was found that simply mixing fine coal particles with water caused aggregation of coal particles in water to impair the fluidity, thus failing to highly load a CWM. This finding suggests that an additive having a high dispersing power is necessary to prevent coal particles from aggregating. Only a few studies (1-6) on this approach demonstrated a relationship between the compositions of additives and the physical properties of CWM.

This report systematically evaluates the ability of surfactants to highly load a CWM and discusses results in terms of the structure of the surfactant. First, surfactants (anionic surfactants, cationic surfactants, nonionic surfactants and amphoteric surfactants) were examined for the ability. Some anionic and nonionic surfactants were found to be effective for highly loading a CWM. Chosen among them were nonionic surfactants containing no alkali metal which would be harmful on combustion. Thus, new types of multi-branched high molecular nonionic surfactants were synthesized; some of them had a greater ability to highly load a CWM. In addition, a relationship between the high loading ability and the surfactant structure was clearly identified.

EXPERIMENTAL PROCEDURES

Materials: Coal. Tatung coal, bituminous coal produced in China, was used. Its analytical values are shown in Table 1.

Materials: Surfactants. Examined in this study were the types of surfactants shown in Table 2 and newly synthesized surfactants described below.

Alcohol base multi-branched high molecular nonionic surfactants. Glycerin, diglycerin and sorbitol of the first reagent-grade were separately used as starting material. Ethylene oxide (EO) made by Mitsubishi Petrochemical Co., Ltd., and propylene oxide (PO) by Asahi Glass Co., Ltd., were added to them. For synthesis, a pressure-resistant stainless steel vessel (5-l capacity) was charged with any one of the above starting materials in such a given quantity that the volume of end product was four l. Potassium hydroxide as catalyst was added to it by 0.1% of end product, and it was dehydrated at a high temperature under vacuum.

Then PO and EO were sequentially added to the starting material in quantities such that a target molar number was attained: First, PO was agitated at 115±5 C under 2 to 2.5 atm and next EO was done at 120±5 C under 1.0 to 1.5 atm, to react with the starting material for 15 to 24 hr in all. Thus, expected surfactants were synthesized. The average molar number of alkylene oxide added was calculated by measuring the OH value by the conventional method. The yield in reaction was 98 to 99%.

Phenolic resin base multi-branched high molecular nonionic surfactants. Phenolic resin of six nuclei [PR(6)], para-secondary butyl phenolic resin of six nuclei [P-SBPR(6)], dodecyl phenolic resin of six nuclei [DPR(6)] and nonyl phenolic resin of four nuclei, six nuclei, 12 nuclei and 19 nuclei [NPR(x)] were used; they had been crosslinked in the presence of sulfuric acid catalyst by the conventional method. The number of nuclei for each was an average value measured by GPC. For

TABLE 1

Analysis of Coal

	Approximate analysis (%)				Ultimate analysis (%)				
Sample	I.M.	Ash	V.M.	F.C.	C	H	O	N	S
Tatung	3.5	10.1	26.6	59.8	84.3	4.8	9.4	0.9	0.6

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TABLE 2

CWM High Loading Ability of Surfactants

Type		Composition	CWM highly loading ability[a]
Anionic surfactants	Salts of carboxylic acids ($-COONa$)	Sodium salts of fatty acids	×
		Sodium salts of arylcarboxylic acids	×
	Sulfates ($-OSO_3Na$)	Sodium salts of alcohol sulfates	×
		Sodium salts of alkylether sulfates	×
		Sodium salts of turkey red oil	×
	Sulfonates ($-SO_3Na$)	Sodium salts of alkylbenzene sulfonates	×
		Sodium salts of naphthalene sulfonates	×
		Sodium salts of formalin condensate of naphthalene sulfonates	△∼○
		Sodium salts of α-olefin sulfonates	×
		Sodium salts of dialkylsulfosuccinates	×
	Phosphates $-OPO_3Na$ $\begin{matrix}-O\\-O\end{matrix}>PO_2Na$	Sodium salts of alcohol phosphates	×
		Sodium salts of alkylether phosphates	×
Cationic surfactants	Primary amine salts ($-NH_2HCl$)	Alkylamine salts	×
		Monoethanolamine salts	×
	Secondary amine salts ($NH \bullet HCl$)	Dialkylamine salts	×
		Diethanolamine salts	×
	Tertiary amine salts ($N \bullet HCl$)	Trialkylamine salts	×
		Triethanolamine salts	×
Cationic surfactants	Quaternary ammonium salts (N^+Cl^-)	Products of tertiary amines and alkyl clorides	×
		Products of tertiary amines and dimethylsulfuric acids	×
Amphoteric surfactants	Amino acids type ($-NHCH_2CH_2COOH$)		×
	Betain type $(-\overset{CH_3}{\underset{CH_3}{N^+}} \bullet CH_2COO^-)$		×
Nonionic surfactants	Polyethylene-grycohol type	Polyoxyethylene alcohol ethers	×
		Polyoxyethylene alkylphenyl ethers	×
		Polyoxyethylene fatty acid esters	×
		Polyoxyethylene polyhydric alcohol fatty acid esters	×
		Polyoxyethylene alkylamines	×
		Polyoxyethylene alkylamides	×
		Polyalkyleneoxide block copolymers	×∼○
	Polyhydric alcohol type	Polyhydric alcohol fatty acid esters	×
		Alkanolamine fatty acid amides	×

[a]×, Mxcc is 60% or less; △, Mxcc is 60%∼65%; ○, Mxcc is 65% or more.

synthesis, the same types of EO and PO and the same procedure as above were used. The yield for the production of expected surfactants was 97 to 98%.

Amine base multi-branched high molecular nonionic surfactants. Diethylenetriamine (DETA) and polyethylene-imine having an average molecular weight of 600 or 1,800 (PEI-600, PEI-1800) were commercially available products. EO and PO were the same as above. For synthesis, a pressure-resistant stainless steel vessel was charged with each starting material, and one mol of PO was added to it in the absence of catalyst. Then potassium hydroxide as catalyst was added to it by 0.1% of end product, and it was dehydrated at high temperature under vacuum. Following that, it was allowed to react with alkylene oxide (AO). The reaction conditions were the same as above. The yield for the production of expected surfactants was 97 to 99%.

Methods: Coal pulverization. Lumps of coal were granulated to less than 5 mm by a Hammer Mill H-12 (made by Hosokawa Micron), and then a stainless steel ball mill (7-l capacity, 50 vol % filled with 32-mm diameter balls) was used to pulverize the coal granules, considering the combustibility of coal, so that 80% of them can pass 200 mesh. Figure 1 shows the particle sizes of pulverized coal.

Methods: Preparation of CWM. A surfactant was precisely measured in such an amount as to be 0.4% coal in the end product. It was well mixed with a given amount of distilled water in a beaker (200 ml). Then, while agitating it at 800 rpm with a three-bladed propeller, pulverized coal particles were added to it incrementally, first gram by gram and then 0.1 g by 0.1 g, and were well mixed. When the slurry viscosity reached 2,000 cp, its coal concentration was measured and defined as maximum coal concentration (MxCC). During mixing, silicone antifoamer was added by 1.0% of the surfactant amount to prevent foaming, or to eliminate an error due to foaming. A slurry viscosity of 2,000 cp was chosen because it generally was said to be an upper limit that allowed a pumping transportation of slurry. In this state, CWM's were obtained with high reproducibility.

The total water content of CWM was determined by the method of JIS M 8811. And the coal concentration of CWM was calculated by this expression: Coal conc (%) = 100 (%) - total water content (%) - surfactant content (%).

Measurement of CWM viscosity. The No. 3 rotor of a Brookfield viscometer was rotated at 60 rpm for one min to measure the viscosity at 25 C.

Methods: Evaluation of CWM stability. To homogeneously disperse the surfactant in a CWM prepared as mentioned above, it was taken by 400 g and further agitated at 3,000 rpm for three min by a lab disperser. A measuring cylinder (500-ml capacity, 5-cm diameter) was filled with this CWM to the 18-cm level, covered with a rubber cap and allowed to stand still at 25 C for the specified number of days. Every seven days, a stainless steel rod (5 mm diameter, 50 g) was dropped vertically from the surface of the CWM to measure rod penetration time (how long it took until the rod reached the bottom).

RESULTS AND DISCUSSION

Ability of general surfactants to highly load a CWM. Generally, the maximum coal concentration of a highly loaded CWM depends on the particle size distribution (7, 8) and the dispersant. That is, an optimum particle size distribution will lead to the highest packing density of coal particles, and a disperant will be able to prevent coal particles from aggregating and allow them to travel freely in water. In this study, which is intended to examine the latter, coal was pulverized by a conventional ball mill and used in a given particle size distribution, as shown in Figure 1.

First, a Tatung coal CWM without surfactant was measured to find that its MxCC was 58.6%. This value means that a highly loaded CWM cannot be produced without surfactant. In the CWM, because coal particles are subject to aggregation in water, with an increase in coal concentration, intervals between coal particles can be reduced to cause rapid aggregation, thus forming a strong crosslinked structure. This structure can impair the fluidity of CWM.

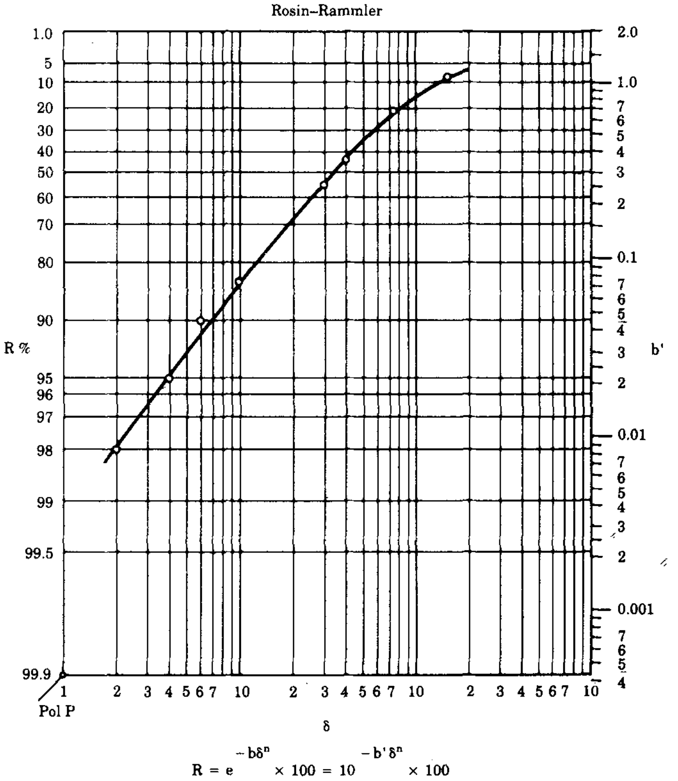

FIG. 1. Particle size distribution of Tatung coal CWM.

Next, to examine how surfactants affected highly loading a CWM, surfactants shown in Table 2 were added by 0.4% vs coal to samples of Tatung coal CWM. Their MxCC values were obtained as shown in Table 2, too. According to the results in this table, an MxCC value of 65% or more was attained with sodium salt of formalin naphthalene-sulfonate condensate having two $-SO_3$ groups or more, among anionic surfactants, or with some types of polyoxyethylene glycol among nonionic surfactants.

All the other surfactants were unable to highly load a CWM up to an MxCC value of 60%.

Then, characteristics at anionic and nonionic surfactants considered to have the ability to highly load a CWM as mentioned above were compared. It was found that the nonionic surfactants were superior to the others in high loading ability; also, they did not contain sulfur or alkali metal which can corrode a boiler. Thus, further experiments were directed mainly toward nonionic surfactants.

Ability of Nonionic Surfactants to Highly Load CWM. Among many types of nonionic surfactant, as shown in Table 2, commonly available types having a low molecular weight of 1,000 or less had no effect on highly loading a CWM, but some types of polyoxyethylene polypropylene glycol having a high molecular weight had good effects. Their ability is detailed in Figure 2, which shows the MxCC values of polypropylene glycol (PPG) having a molecular weight of 1,200, 2,000 or 3,200, terminated with ethylene oxide (EO) by the varied numbers of mols. Compared with when the molecular weight was 1,200 or 2,000, EO-added PPG of 3,200 molecular weight yielded better results, making the best MxCC value, about 68%. It was also found that the larger the number of mols of added EO was, the better the high loading ability was.

From the results of the above paragraph, it was presumed that surfactants with higher molecular weight and more massive molecular structure would have better ability to highly load a CWM.

Thus, surfactants with alkylene oxide (AO) added by the larger number of mols and those with the larger number of side chains were examined for the ability. First, multi-branched adducts of polyhydric alcohol such as glycerin (Gl), diglycerin (Digl) or sorbitol (So) with AO were synthesized to measure the MxCC values.

The surfactants of Gl, Digl or So with EO yielded MxCC values of less than 60%, having no great effect on a CWM. Their results are omitted here because they are not important.

Next, the adducts of Gl, Digl or So with PO-EO were investigated. To discuss their ability for high loading and the steric property of their molecules, figures were plotted with MxCC values on the Y axis and AO molecular weight per active hydrogen atom related to chain length on the X axis. Figure 3 is for the adduct of Gl with PO-EO. This figure shows that the adduct is excellent in the ability, giving the best MxCC value of about 69%, and that the MxCC value tends to be larger with an increase in molecular weight per active hydrogen atom, reaching its maximum at a molecular weight of 6,000 or more. The number of mols of PO added is preferred to be 120 or less; 60 is particularly desirable. The larger number of mols causes the MxCC value to be reduced. These results suggest that a surfactant for CWM should contain side chains having a molecular weight of 6,000 or more and PO and EO in an appropriate ratio.

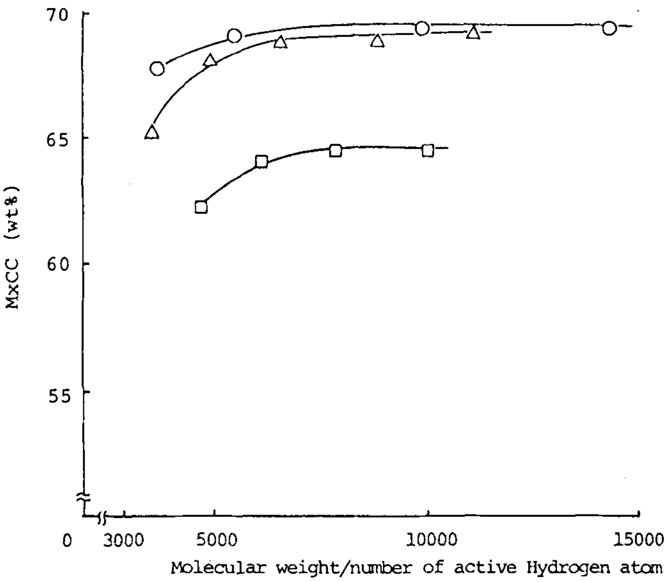

FIG. 3. MxCC value of polyoxyethylene-polyoxypropylene glycerol ethers. ○, Gl $PO_{60}EO_x$; △, Gl $PO_{120}EO_x$; □, Gl $PO_{180}EO_x$.

Figure 4 is for the adduct of Digl with PO-EO. It indicates that this adduct is also excellent in the ability, and that the MxCC value can be up to about 69% when the adduct is produced by addition copolymerization of PO and EO in a proper ratio so that the molecular weight of its side chain is 6,000 or more.

The results of the adduct of So with PO-EO are shown in Figure 5. As in the above results, this adduct can also make the MxCC value up to about 69%, and when the molecular weight per active hydrogen atom, or the molecular weight of side chain, is 6,000 or more, its ability to highly load a CWM is best.

From the results of Figures 3, 4 and 5, to impart a

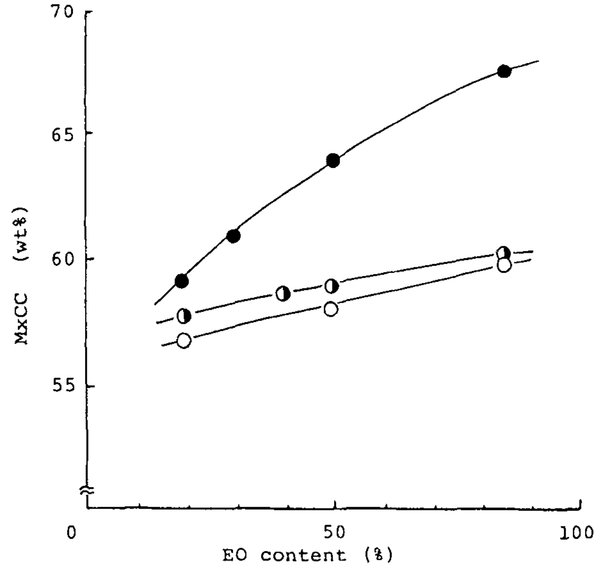

FIG. 2. MxCC value of polyoxypropylene polyoxyethylene block copolymer. Starting substance: ○, PPG 1200; ◑, PPG 2000; ●, PPG 3200.

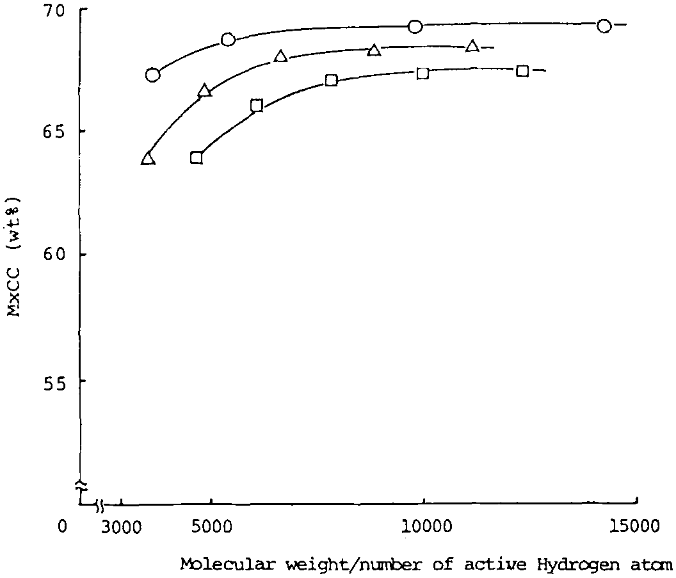

FIG. 4. MxCC value of polyoxyethylene-polyoxypropylene diglycerol ethers. ○, Digl $PO_{80}EO_x$; △, Digl $PO_{160}EO_x$; □, Digl $PO_{240}EO_x$.

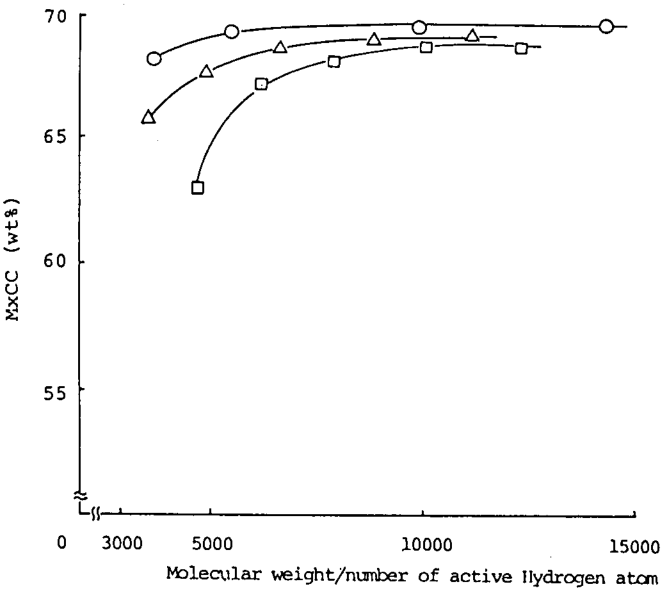

FIG. 5. MxCC value of polyoxyethylene-polyoxypropylene sorbitol ethers. ○, So $PO_{120}EO_x$; △, So $PO_{240}EO_x$; □, So $PO_{360}EO_x$.

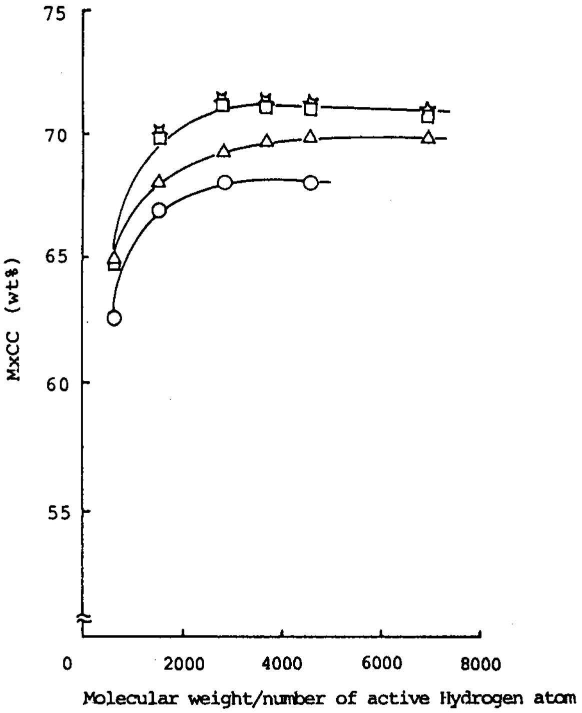

FIG. 7. MxCC value of polyoxyethylene nonylphenyl resin. ○, NPR (4) EO_x; △, NPR (6) EO_x; □, NPR (12) EO_x; ☆, NPR (19) EO_x.

good high loading ability to alcohol-based multibranched high molecular nonionic surfactants, it is necessary to keep the PO-to-EO ratio appropriate and to make the molecular weight of the side chain 6,000 or more. A starting material is allowed to be of Gl, Digl or So. In addition, it is noted that the number of active hydrogen atoms for the adduct of PPG with EO discussed earlier is compared with that for the adducts of polyhydric alcohol. That is, the former is 2, but the latter is 3, 4 and 6 for Gl, Digl and So, respectively. This difference in the number of branches contributes to an increase in the MxCC value.

As briefly described, when polyhydric alcohol was used as the starting material, adducts with only EO were very inferior in high loading ability. The polyhydric alcohol is a very hydrophilic compound, and thus the addition of only EO to it does not provide any lipophilic part or allow it to function as a surfactant. This fact probably caused the unfavorable results. Because the surfaces of coal particles in a coal-water system are relatively lipophilic, a surfactant for it probably needs to be so lipophilic as to have effects on the coal-water interface. For the adducts with PO-EO, the smaller the number of mols of PO added is, the better the high loading ability is, as mentioned earlier. This suggests that only a little lipophilic part is required for such surfactants.

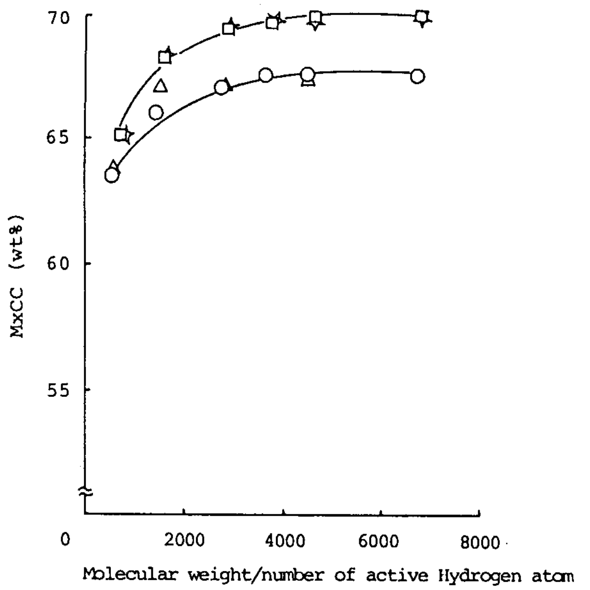

FIG. 6. MxCC value of polyoxyethylene alkylphenyl resin. ○, PR (6) EO_x; △, PSBPR (6) EO_x; □, NPR (6) EO_x; ☆, DPR (6) EO_x.

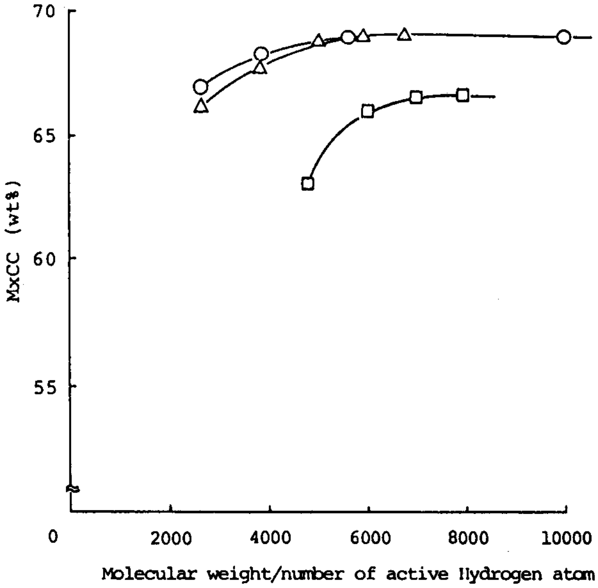

FIG. 8. MxCC value of polyoxyethylene-polyoxypropylene diethylene triamine ethers. ○, DETA $PO_{100}EO_x$; △, DETA $PO_{200}EO_x$; □, DETA $PO_{300}EO_x$.

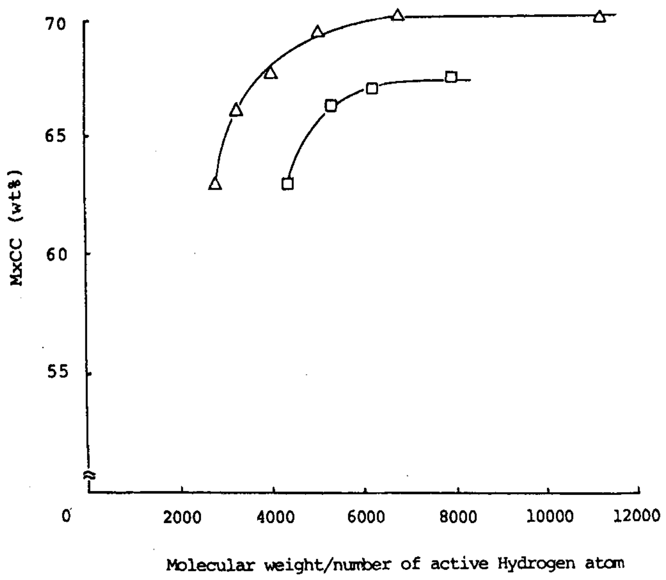

FIG. 9. MxCC value of polyoxyethylene-polyoxypropylene polyethylenepolyamine (MW 600) ethers. Δ, **PEI (600) $PO_{560}EO_x$;** □, **PEI (600) $PO_{840}EO_x$.**

Resins of a lipophilic group, which can have more active hydrogen atoms than polyhydric alcohol, were used as starting material to synthesize surfactants with EO added. That is, such surfactants were enhanced in the degree of branching by crosslinking phenol or alkylphenol with formalin and were made high in molecular weight with EO added by the large number of mols. First examined was the high loading ability of phenol resin base multi-branched high molecular nonionic surfactants with different side-chain alkyl groups. With such surfactants with the same number of nuclei and the different numbers of mols of EO added, MxCC values were measured as shown in Figure 6. To illustrate the relationship between the steric property of molecules and their high loading ability, this figure also shows the molecular weight per active hydrogen atom on the X axis. The adducts of PR(6) with EO and of P-SBPR(6) with EO are similar in the ability; the MxCC value is larger with an increase in molecular weight per active hydrogen atom, reaches about 67% at about 3,000 and keeps the level at more than 3,000. The adducts of NPR(6) with EO and of DPR(6) with EO are better in the ability than the former two, making the MxCC value constant at about 70% when the molecular weight per active hydrogen atom is 3,000 or more. These results indicate that the nonyl or dodecyl group is more effective for CWM than the butyl group, and that the molecular weight per active hydrogen atom is required to be 3,000 or more.

Next, to discuss the relationship between the degree of crosslinking and the high loading ability, surfactants were synthesized by adding EO to NRP, varying the number of nuclei. And they were evaluated for the high loading ability as shown in Figure 7. From this figure, it is found that the number of nuclei for NPR is preferably 12 to 19, but has unfavorable effects at 4. The molecular weight per active hydrogen atom should be 3,000 or more for the best ability, regardless of the number of nuclei.

As evidenced above, steric and massive multibranched high molecular nonionic surfactants have the ability to highly load a CWM. The authors tried to develop this to make a more massive structure than in the above-mentioned surfactants. Thus, amine base multibranched high molecular nonionic surfactants were synthesized by adding PO-EO to PEI(600), PEI(1800) or DETA as starting material, and their high loading ability was evaluated.

Figure 8 shows the relationship between the MxCC value and the molecular weight per active hydrogen atom for the adduct of DETA with PO-EO. The number of mols of PO added was 20, 40 or 60, as for polyhydric alcohol surfactants, and after that, EO was added. This figure indicates that the MxCC value is

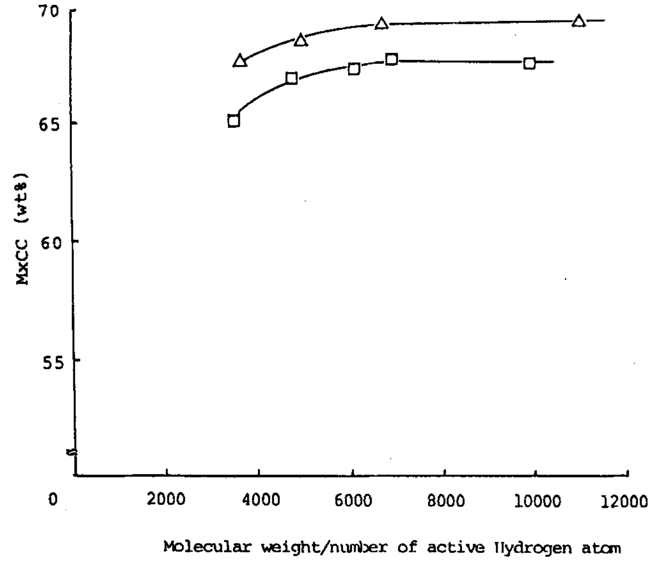

FIG. 10. MxCC value of polyoxyethylene-polyoxypropylene polyethylenepolyamine (MW 1800) ethers. Δ, **PEI (1800) $PO_{1680}EO_x$;** □, **PEI (1800) $PO_{2520}EO_x$.**

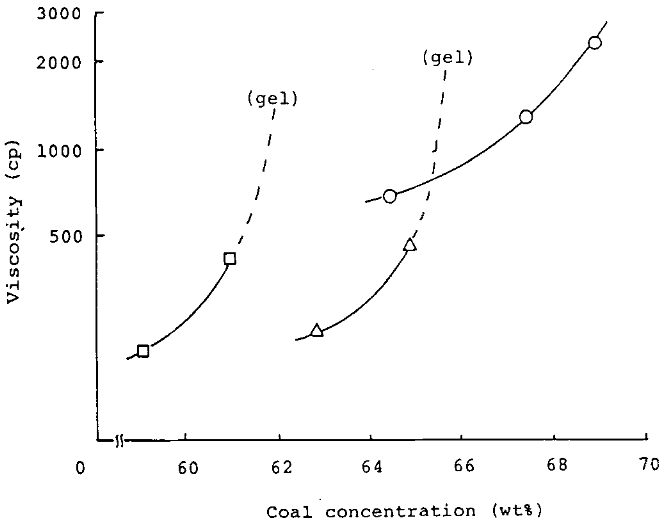

FIG. 11. Relationship between coal concentration and slurry viscosity in Tatung Coal CWM. ○ **PEI (600) $PO_{560}EO_{1400}$ 0.4% (vs coal);** Δ, **NSF-Na 0.4% (vs coal);** □, **LS-Na 0.4% (vs coal).**

TABLE 3

Stability of Tatung Coal CWM[a]

	CWM properties		Rod penetration time (sec)			
Surfactants[b]	Coal concentration (%)	Viscosity (cp)	After 7 days	After 14 days	After 21 days	After 28 days
$SoPO_{120}EO_{600}$	67.0	1200	1.2	1.5	2.0	3.5
$NPR(12)\ EO_{1200}$	67.0	1120	1.2	1.4	2.2	3.8
$PEI(600)\ PO_{560}EO_{1400}$	67.0	1000	1.0	1.2	1.8	2.2

[a]Particles passing 200 mesh, 80%.

[b]Adding amount, 1.0% (vs coal).

larger with an increase in the molecular weight and reaches its maximum at about 6,000. The number of mols of PO added is optimum at 100 (20 mols/active hydrogen atom) or 200 (40 mols/active hydrogen atom). When it is too large, the MxCC value is low.

Figure 9 and 10 are for the adducts with PO-EO of PEI(600) and of PEI (1,800), respectively. These surfactants also make the MxCC value larger, with an increase in the molecular weight, and largest at about 6,000. Adding PO by 40 mols/active hydrogen atom imparts the best effects. That is, the surfactant of $PEI(600)PO_{560}EO_{1400}$ exhibits the highest ability, making an MxCC value of 70%.

From Figures 8, 9 and 10 it is found that the molecular weight of starting material is optimum at about 600, and that the high loading ability is maximized, though a little difference is found between surfactants, when the molecular weight per active hydrogen atom is about 6,000, and is kept maximum at more than 6,000. These surfactants, designed to have a steric and massive structure of lipophilic property, are proved to exhibit their best dispersing power.

We have discussed the high loading ability of multi-branched high molecular nonionic surfactants with polyhydric alcohol, phenolic resin or amine as starting material. As a result, it is found that every one of those surfactants tends to improve its high loading ability as the molecular weight/active hydrogen atom is increased, and that the ability reaches its maximum at the molecular weight of about 6,000 for polyhydric alcohol base, at about 3,000 for phenolic resin base, or at about 6,000 for amine base. The reason for this difference is probably that such surfactants form micelles with the lipophilic group facing inward and the hydrophilic group facing outward, in water. In a phenol resin system, since its lipophilic group is very lipophilic, its micelle has lipophilic and hydrophilic groups clearly facing inward and outward, respectively. In a phenol resin base surfactant whose molecular weight per active hydrogen atom is 3,000, the length of its micelle is supposed to be equivalent to a molecular weight of 6,000 (3,000 × 2). On the other hand, in a system of polyhydric alcohol or amine, since its lipophilic group, PO, is not so high in lipophilic property as that of phenolic resin, its micelle cannot so clearly face the lipophilic group inward. Therefore, a surfactant of polyhydric alcohol or amine needs the molecular weight of 6,000 equivalent to the micelle length which is presumed to be required from the results of a phenolic resin base surfactant.

Properties of CWM. Among multi-branched high molecular nonionic surfactants studied, the $PEI(600)PO_{560}EO_{1400}$ exhibiting the best high loading ability was taken to make CWM samples at varied coal concentrations. Their viscosity was measured as shown in Figure 11. At the same time, for comparison, sodium salt of formalin naphthalenesulfonate condensate (NSF-Na), which is proved to be effective, and sodium lignosulfonate (Ls-Na) were used as anionic surfactants to make CWM samples at varied coal concentrations. Their viscosity was also measured as shown in Figure 1.

This figure indicates that the $PEI(600)PO_{560}EO_{1400}$, when added by 0.4% vs coal, can allow a CWM to have a coal concentration of 68% and a viscosity of 1500 cp. For NSF-Na, when added by 0.4% vs coal, a CWM of 65% and 500 cp can be obtained, but at higher coal concentrations it abruptly thickens to gel. Ls-Na can only load a CWM with up to 61% coal, and at higher coal concentrations it also thickens abruptly. These results suggest that anionic surfactants are inferior in the highly loading property to multi-branched high molecular nonionic surfactants. It is probably because the former is lower in molecular weight and less steric and massive than the latter, thus lacking the power to prevent coal particles from being aggregated.

Stability of CWM. To put a CWM to practical use, its additive must have not only the above-mentioned high loading ability but also a stabilizing power to prevent coal particles from settling. Tatung coal CWM samples were prepared with surfactants having yielded good results such as $SOPO_{120}EO_{600}$, $NPR(12)EO_{1200}$, or $PEI(600)PO_{560}EO_{1400}$, added by 1.0% vs coal. Their stability was evaluated as shown in Table 3. With any of these surfactants, almost no coal settling was found after a 28-day storage, indicating that they are also excellent in stabilizing power and will be useful for practical production of CWM.

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