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FLOATABILITIES OF TREATED COAL IN WATER AT ROOM TEMPERATURE

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ABSTRACT

Experiments on equilibrium adsorption loadings of various probe compounds on 60-200 mesh Illinois #6 coal (PSOC-1539), Adaville #1 coal (PSOC-1544), Wyodak coal (PSOC-1545), and Pittsburgh #8 coal (PSOC-1549) were performed. The probe compounds include 2-methyl-1-pentanol (2M1P), 1-heptanol, benzene, and toluene. Equilibrium adsorption loadings of aromatic compounds such as toluene and benzene on the four chosen coals obey the Langmuir isotherm model up to 100 ppm in concentrations of probe compounds. Equilibrium adsorption loadings of higher aliphatic alcohols such as 2M1P and 1-heptanol on the four chosen coals do not follow both the Langmuir isotherm model and the Freundlich empirical adsorption model. Flotation of the coals, equilibrated with aqueous solutions of 2M1P and 1-heptanol, increases linearly with equilibrium adsorption loadings of these probe compounds on the coals.

The chosen coals were treated with nitrogen and air at 1 atm and 125-225°C for 24 h. Flotation experiments of the treated coals were conducted at room temperature, using distilled water only as a flotation medium. Flotation of Adaville #1 coal and Wyodak coal treated with nitrogen gas is higher than that of the untreated coals and increases with treatment temperatures. Flotation of Adaville #1 coal treated with air at 125-225°C is not significantly different from that of untreated coal. Flotation of Pittsburgh #8 coal treated with air is lower than that of untreated coal and decreases with treatment temperatures. Flotation of Illinois #6 coal treated with nitrogen only is higher than that of untreated coal. Flotation of Illinois #6 coal treated with nitrogen at 125-175°C increases with treatment temperatures, whereas flotation of Illinois #6 coal treated with nitrogen at 175-225°C decreases with treatment temperatures.

INTRODUCTION

Coal is the most plentiful energy resource in the United States and continues to be the major fuel utilized by electrical power plants. The coal resources will become more attractive for chemical feedstocks, as well as sources of liquid and gaseous fuels, as petroleum resources become more depleted.

Raw coal consists of minerals and organic matter. Coal minerals include primarily pyrites, silicates (quartz, clays), and various minor minerals; whereas organic matter is composed of carbon, oxygen, hydrogen, nitrogen, and sulfur.

Sulfur in coal is present as organic sulfur, pyritic sulfur, and sulfate sulfur. Sulfur occurring in the form of relatively coarse pyrite particles is removed largely by density separation, but very finely disseminated pyrite and organic sulfur cannot be removed in this way. Sulfur products in combustion not only pollute the environment, but they also are detrimental to combustion systems because of the corrosive action of their combustion products. Sulfur is a harmful impurity in all metallurgical products.

The ash-forming impurities in coal not only dilute the combustible contents of the coal, and thus decrease its heating value, but also reduce the effective capacity of the burning equipment. Minerals cause solid buildup and corrosion on boiler surfaces, due principally to alkali metals and alkali chlorides from clay minerals.

Coal is a very complex and heterogeneous material. Effective utilization of coal requires an understanding of its formation, structure, and purification. Extensive research of physical and chemical coal cleaning is being carried out to increase the heating value of coal and reduce pollution of the environment by sulfur oxides, nitrogen oxides, and toxic metals. Various additives are used in cleaning coal with water to modify surface properties of coal and enhance separation of minerals from coal particles.

Several methods of analyzing surface properties of raw coal and treated coal have been employed. These include adsorption isotherms, electrokinetics data, contact angle measurements, and floatabilities. Recently, many researchers utilize calorimeters to measure heat of interaction at the liquid-solid interfaces. More recently, the inverse gas chromatography technique was employed to analyze surface properties of various coals using probe gaseous compounds as adsorbates. The inverse liquid chromatography technique and the flotation method are applied to evaluating surface properties of raw coals and various treated coals in this study.

The main objectives of this research are to refine further the inverse liquid chromatography technique in evaluating surface properties of raw coals, to investigate mechanisms on adsorption of probe compounds on various coals, and study changes in surface properties of treated coals by evaluating floatabilities of treated coals in water only as a flotation medium.

Coals such as Pittsburgh #8 coal, Illinois #6 coal, Adaville #1 coal, and Smith-Roland Wyodak coal for this research are chosen as representatives of high-rank bituminous coal, high-volatility bituminous coals, and subbituminous coal, respectively. Aliphatic alcohols such as 2-methyl-1-pentanol and 1-heptanol are utilized as probe compounds to investigate floatability of the coals, equilibrated with their aqueous solutions at room temperature. Aromatic compounds such as benzene and toluene are used in studying mechanisms of their adsorption on the coals.

The chosen coals were treated with nitrogen and air at 125-225°C. Experiments on flotation of treated coals were conducted in a batch-type flotation apparatus at room temperature, using water only as a flotation medium, to investigate changes in surface properties of the treated coals.

BACKGROUND

All coal cleaning processes in general use are gravity-concentration methods. Methods based upon surface properties are employed for further beneficiation, especially for fine coals, in order to clean coal to a level that is acceptable to the strict air pollution regulations. The froth-flotation process depends on hydrophobicity differences between the surface of coal and those of its coal mineral impurities, using water as a liquid medium. The froth flotation process is independent of specific gravity and depends almost completely on the surface properties of coal.

Chemical technology is especially applicable to the flotation process because it depends on the surface properties of the coal that can be physically modified in the presence of reagents such as frothing agents, collecting agents, and depressing agents (1) or even altered completely by treating coal with appropriate chemicals.

High-rank bituminous coals are normally floatable in the presence of a frothier without collectors and pulp conditioning. However, low-rank bituminous coals, anthracite, and oxidized coals may require a collector and a reagent conditioner to render the coal particles hydrophobic. Ordinarily, a nonpolar oil such as kerosene or fuel oil is used along with a frothier (2).

Pierters (3) conducted laboratory experiments with various flotation reagents. Both organic acids and alcohols were found to increase their effectiveness in the coal flotation process with high molecular weights. A series of mineral oils showed increased collecting power with decreased viscosity of oil. Phenol was strongly affected by changes in pH.

Coal floatability is dependent on the oxidation state of a coal, its surface polarity, and coal rank. The significant oxygen-containing functional groups in coal are carbonyl, hydroxyl, carboxyl, and methoxyl (4). The high concentration of surface polar groups results in a reduced surface hydrophobicity and hence a lessened floatability during the flotation experiments. Coal is an extremely reactive material toward oxidation. Even small amounts of air (oxygen) can significantly alter the physical and chemical properties of coal (5). Oxidation of the coal surface results in an adverse effect on its floatability (6). An oxidized coal tends to be more hydrophilic.

The relative hydrophobicity of a given coal can be evaluated by measuring coal floatability and heats of immersion of coal into water. The coals of various ranks and the solid minerals associated with the coals were characterized in terms of relative hydrophobicity through the values of heats of immersions of the coals in water and in methanol. Heats of immersion of coal were also measured in surfactant solutions of varying concentrations. Heats of immersion of coal were found to correlate well with coal floatability (7).

In pursuit of a relatively sensitive and convenient method for the study on surface properties of coal, the so-called inverse liquid chromatography technique was applied to the evaluation of surface properties of coal in this research study.

The main advantage of the inverse liquid chromatography technique is to easily adjust amounts of coal for an experimental run by varying sizes of packing columns. In this way, minute adsorption of adsorbates on coal surface can be detected easily. Consequently, infinitesimal change in surface properties of coal, which can not be detectable with other methods, such as contact angle measurements, electrokinetics data, and microcalorimetry, is possibly identified with the inverse chromatography technique. In-situ treatments of coal in a column save experimental times and provide convenience in handling coal samples during experimental runs (8).

Another advantage of the inverse liquid chromatography technique, using water as a carrier fluid, appears to be more practical and realistic in understanding surface properties of coal for the beneficiation of coal than the inverse gas chromatography technique, since water is mainly utilized in coal beneficiation processes such as froth-flotation separation, oil agglomeration separation, and gravity separation (8).

Numerous investigators (9, 10) employed the inverse gas chromatography technique to study surface properties of polymers, chromosorbs, porapak Q, and chemically treated compounds such as silicas and asphalts using probe compounds. The surface properties of silicas modified with alcohols were investigated with probe compounds by inverse gas chromatography. The surface properties, such as standard free energies of adsorption, surface energies, and enthalpies of adsorption, were determined with retention volumes from inverse chromatography at various temperatures. The probe chemicals include alcohols such as methanol, ethanol and butanol; alkanes with the carbon number 6 through 10; ether; chloroform; and benzene (11).

More recently, several researchers (12, 13, 14) attempted to utilize this technique in the study of surface properties of coal such as reactivity, surface energy, polarity, softening point, and penetration of probe compounds into the coal structure.

The properties of lignites were studied by a molecular probe technique, using probe compounds such as air, carbon dioxide, water, methanol, and hexane. The probe molecules are adsorbed on the surface of lignite by an adsorption mechanism alone up to 175°C, irrespective of their chemical identity. Water and methanol adsorbed on active hydrophilic sites in monolayer or submonolayer coverage can only be eluted easily around 175°C. The column permeability appears to depend on the sulfur content of lignites (12).

A 28 x 0 mesh, Illinois #6 high-volatility bituminous coal was packed as a packing material in an inverse gas-chromatographic column. The probe compounds include methane, pentane, hexane, cyclohexane, benzene, methanol, propanol, pyridine, 2,4-dimethylphenol, and O-ethylphenol to study changes in coal reactivity as a function of various pretreatments. No major shift in methane's retention time or change in peak shape occurred until after the column temperature was raised above 215°C. At higher temperatures, the methane retention times increased and the peak shapes broadened. This observation may be due to irreversible changes in the coal structure (13).

Asphalts were reacted with N, O-bis (trimethylsilyl)acetamide (BSA) and hexamethyldisilazane (HMDS) at 160°C. Silylation of asphalts within inverse gas-liquid chromatographic columns offers a convenient means to study asphalt functional groups, such as phenolic and carboxylic acid OH functional groups, using probe compounds such as phenol and propionic acid. Asphalt contains many types of functional groups that were found to exhibit selective affinities for specific probe compounds by calculating specific interaction coefficients with retention volume data of probes from inverse chromatography (14).

In the same way, surface properties of coal were evaluated by choosing proper probe compounds and proper operation conditions of a chromatograph in this study, using the inverse liquid chromatography technique.

EXPERIMENTAL

Experiments on equilibrium adsorption loadings of aromatic compounds such as benzene and toluene on Illinois #6 coal, Adaville #1 coal, Smith-Roland Wyodak coal, and Pittsburgh #8 coal were performed in aqueous solutions at room temperature.

Experiments on equilibrium adsorption loadings of aliphatic alcohols such as 2-methyl-1-pentanol and 1-heptanol on Illinois #6 coal, Adaville #1 coal, Smith-Roland Wyodak coal, and Pittsburgh #8 coal were performed in aqueous solutions at room temperature to relate adsorption loadings of these compounds to flotation percentages of these coals equilibrated with their aqueous solutions at room temperature.

The coals were pulverized and sieved to obtain 60-200 mesh coal particles. Sieved 60-200 mesh coal particles were washed with distilled water (DW) to remove water-soluble organic compounds from the surfaces of the coal particles. Coal particles were submerged under water overnight and then filtered the next day to remove organic-dissolved water from surfaces of coal particles. These washing processes continued for several weeks. Washed coal particles were dried at 110°C for 4 h.

Dry coal particles of 0.75 g were introduced into a 0.43-cm-ID, 25-cm-long empty column for experiments on equilibrium adsorption loadings of probe compounds on the coals. The coal particles packed in the column were washed with DW for 10 h to eliminate any water-soluble contaminants introduced in the coal-packed column during the process of packing dry coal particles into the column. Teflon particles also were packed in the empty column to obtain a reference column.

An aqueous solution of a probe compound was fed into the coal-packed column at 0.5 cm^3/min, using a liquid pump. Equilibrium adsorption loadings of probe compounds on coal were obtained from their concentration differences between the coal-packed column and the reference column at room temperature, where concentrations of probe compounds in aqueous solutions were detected with a refractive index detector. Experiments on flotation of the coals were carried on with a 500-cm^3 batch-type micro-flotation apparatus at room temperature. Nitrogen gas was introduced at the bottom of the flotation apparatus.

An empty column was filled with 2.5 g 60-200 mesh coal and then placed in an oven to be treated with nitrogen and air for 24 h at 125-225°C. Flotation experiments of the treated coals also were conducted in the batch-type micro flotation apparatus with nitrogen gas introduced at the bottom of the flotation apparatus at room temperature, using distilled water only as a flotation medium.

DISCUSSION

Equilibrium adsorption loadings of benzene are lower than those of toluene on the four chosen coals (see Figures 1 and 2). Two different adsorption mechanisms are chosen to explain these observations. The first one is based on the reactivity of benzene, and the second one is based on the steric hinderance. The hydrophobic adsorption mechanism is ruled out, since the solubilities of both compounds in water are almost same at room temperature.

In the first adsorption mechanism, toluene is formed by substituting one hydrogen of benzene with a methyl group. The substituted methyl group causes the benzene ring of toluene to be more reactive than benzene. In this way, interactions between toluene and coal may be higher than those between benzene and coal, where benzene ring is assumed to be adsorbed on these coals.

In the second adsorption mechanism, toluene may be adsorbed on these coals by the association of the methyl group of toluene with functional groups of these coals, whereas benzene may be adsorbed on these coals by the association of the CH_2 groups of benzene with functional groups of these coals. Effects of steric hinderance may be dominant in the adsorption of these aromatic compounds on these coals.

Equilibrium adsorption loadings of both benzene and toluene on Adaville #1 coal and Wyodak coal are higher than those on Illinois #6 coal and Pittsburgh #8 coal. Both Adaville #1 coal and Wyodak coal contain 14 wt % oxygen, whereas both Illinois #6 coal and Pittsburgh #8 coal contain 8 wt %

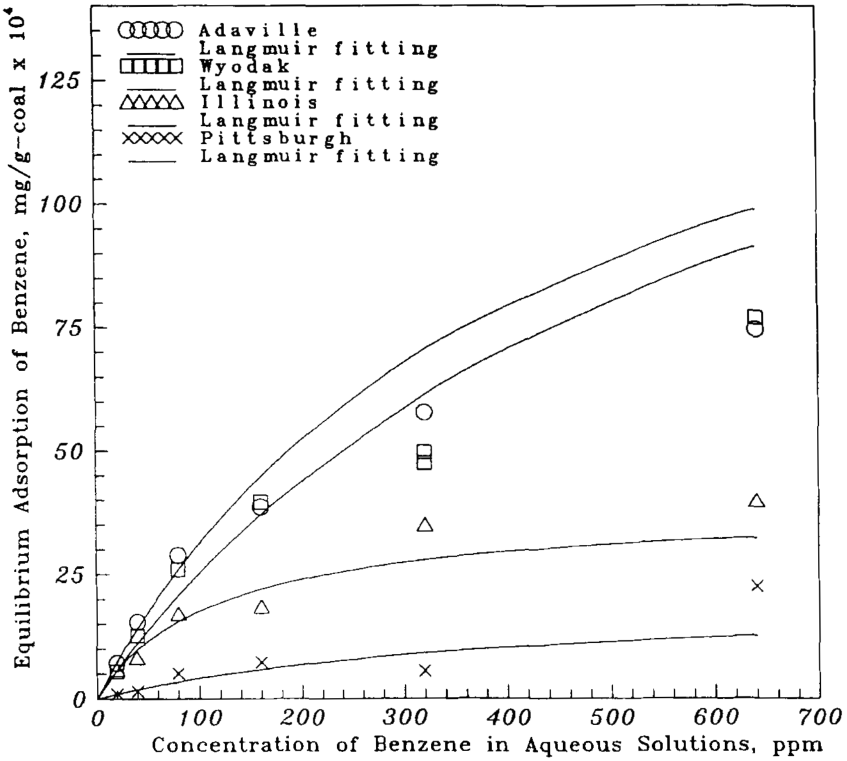

FIGURE 1. Equilibrium adsorption of benzene on 0.75-g 60-200 mesh coal in aqueous solutions at room temperature.

oxygen (see Table 1). Benzene and toluene appear to be adsorbed on these coals by the association with oxygen-containing functional groups such as phenolic hydroxyl groups, carboxylic groups and carbonyl groups of these coals.

Equilibrium adsorption loadings of aromatic compounds such as toluene and benzene on the four chosen coals obey the Langmuir isotherm model up to 100 ppm in concentrations of probe compounds (see Figures 1 and 2). Equilibrium adsorption loadings of both benzene and toluene on both Wyodak coal

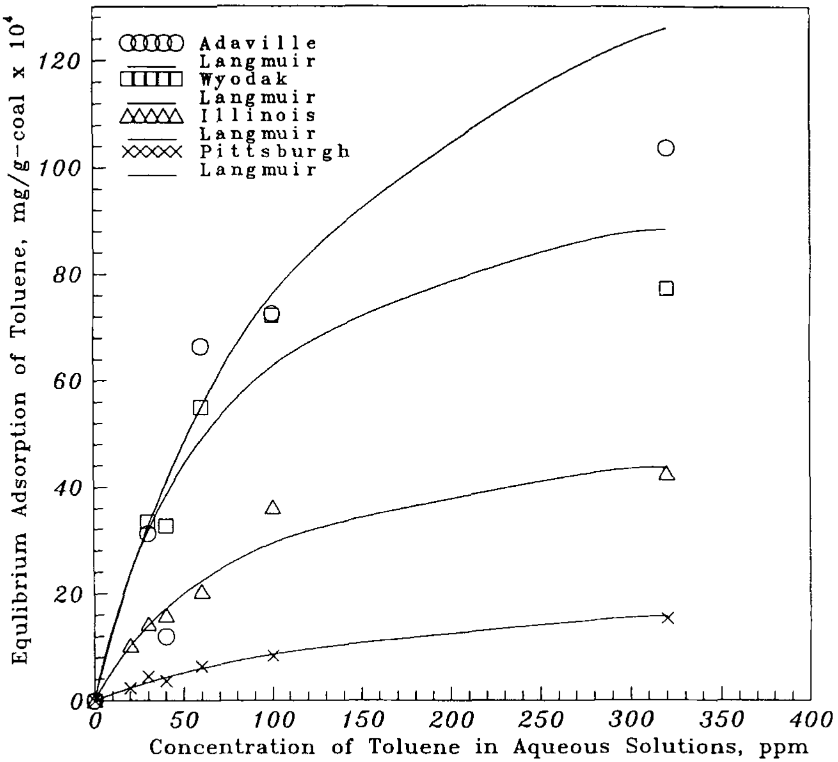

FIGURE 2. Equilibrium adsorption of toluene on 0.75-g 60-200 mesh coal in aqueous solutions at room temperature.

and Adaville #1 coal are overpredicted by the Langmuir isotherm model at relatively high concentrations of these compounds. These observations may indicate that both Wyodak coal and Adaville #1 coal swell in the presence of these aromatic compounds, and surface pores accessible to these compounds collapse in the presence of these aromatic compounds at relative high concentrations.

Equilibrium adsorption loadings of both 2-methyl-1-pentanol (2M1P) and 1-heptanol on both Adaville #1 coal and Wyodak coal are higher than those on

Table 1. Ultimate Analyses of Various Coals (dry basis), w %

PSU #	PSOC-1539	PSOC-1544	PSOC-1545	PSOC-1549
Seam Name	Illinois #6	Adaville #1	Wyodak	Pittsburgh #8
Coal Rank	HVC	HVC	Subbit C	HVA
Ash	16	4	14	10
Carbon	65	74	64	75
Hydrogen	5	5	5	5
Nitrogen	1	1	1	1
Sulfur	5	1	1	1
Oxygen	8	14	14	7

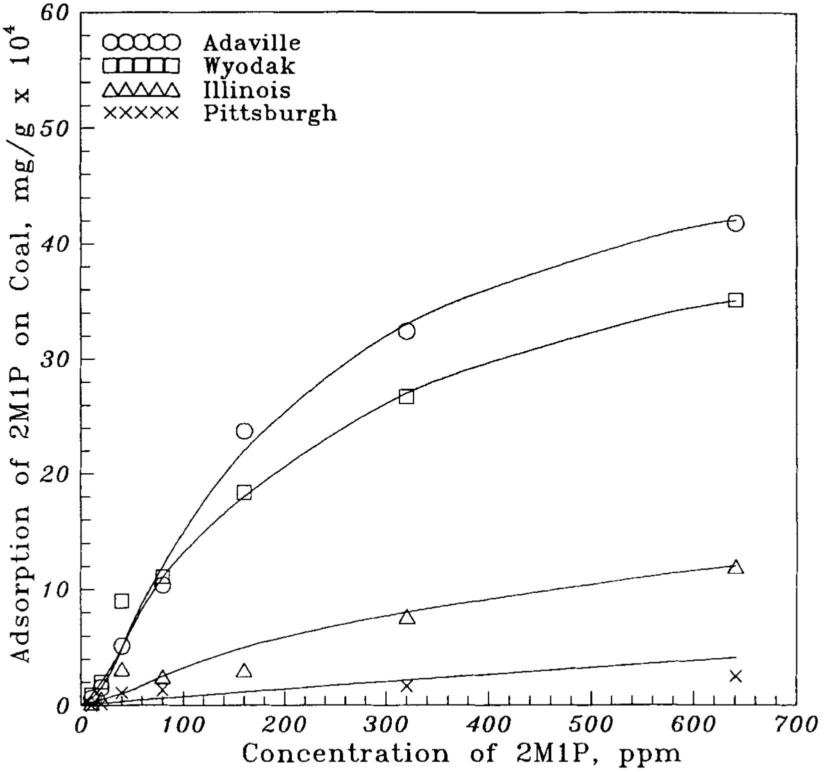

FIGURE 3. Equilibrium adsorption of 2-methyl-1-pentanol (2M1P) on 0.75-g 60-200 mesh coal in aqueous solutions at room temperature.

both Illinois #6 coal and Pittsburgh #8 coal (see Figures 3 and 6), as shown in equilibrium adsorption loadings of both toluene and benzene on these coals. These data may indicate that both Adaville #1 coal and Wyodak coal contain more hydrophilic functional groups than both Illinois #6 coal and Pittsburgh #8 coal.

Equilibrium adsorption loadings of 1-heptanol on the four chosen coals are higher than those of 2M1P on these coals. The solubility of 1-heptanol in

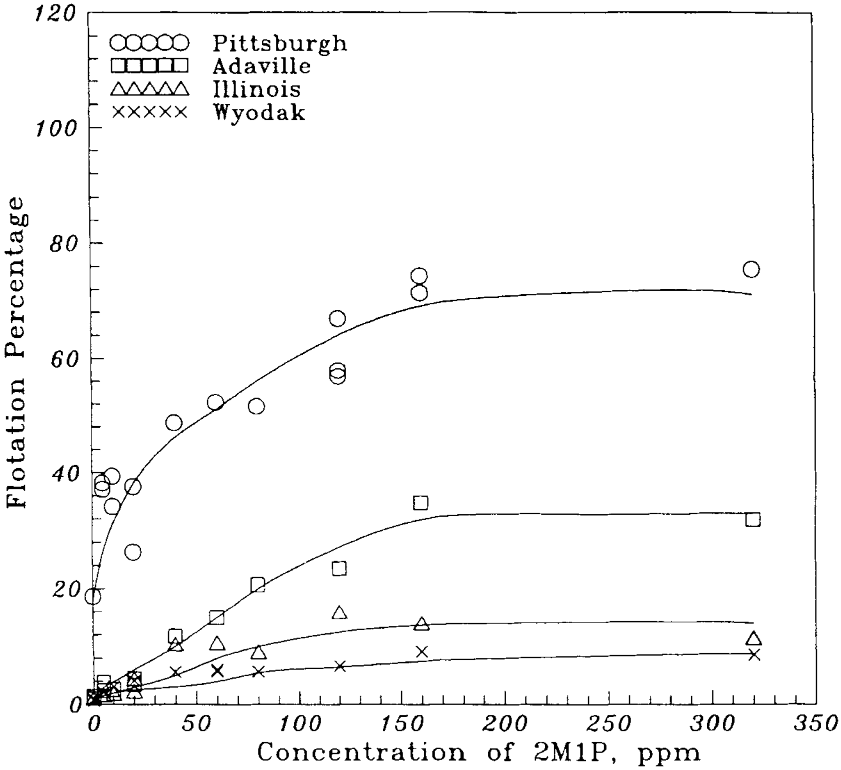

FIGURE 4. Flotation of various coals, equilibrated with 2-methyl-1-pentanol (2M1P) at room temperature.

water is lower than that of 2M1P in water. Reactivity of hydroxyl groups for aliphatic alcohols decreases with increased number of carbons of aliphatic alcohols. Aliphatic alcohols with branched carbon chains are less reactive than those with straight carbon chains. Solubility of aliphatic alcohols appears to affect significantly their adsorption on coal in addition to surface properties of coal. Hydrophobic adsorption mechanism appears to be applicable to explaining these observations.

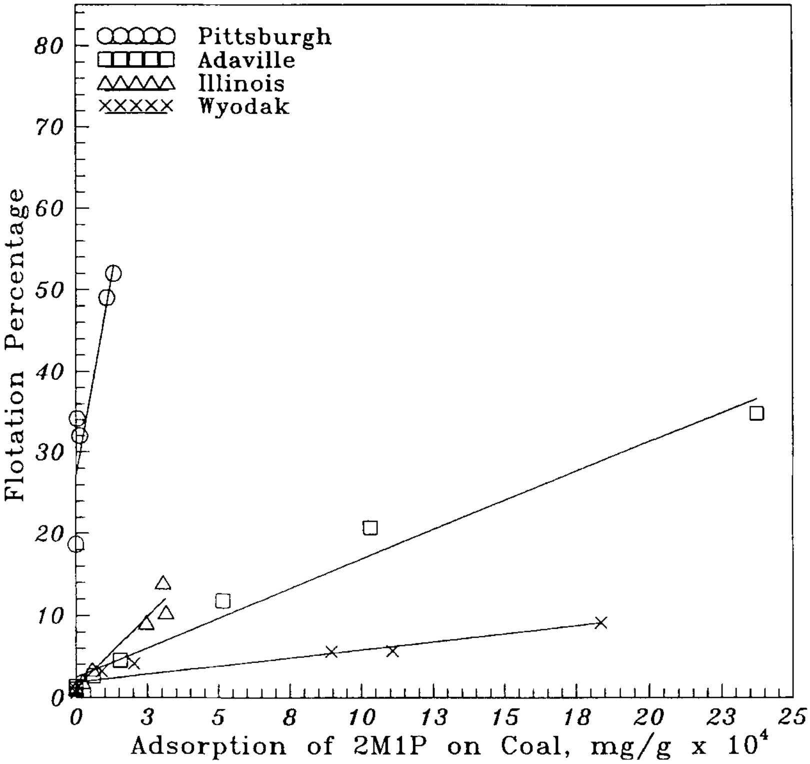

FIGURE 5. Flotation vs. adsorption of 2-methyl-1-pentanol (2M1P) for various coals at room temperature.

Flotation of Pittsburgh #8 coal with aqueous solutions of both 2M1P and 1-heptanol is highest among the coals, and flotation of Wyodak coal is lowest among the coals (see Figures 4 and 7). These facts also show that Pittsburgh #8 coal is more hydrophobic than Wyodak coal. Flotation of both Pittsburgh #8 coal and Adaville #1 coal is higher in the presence of 1-heptanol than 2M1P. These observations may suggest that water affinity of the compounds adsorbed on the coals affects more significantly floatability of the coals in water than molecular structure of the compounds.

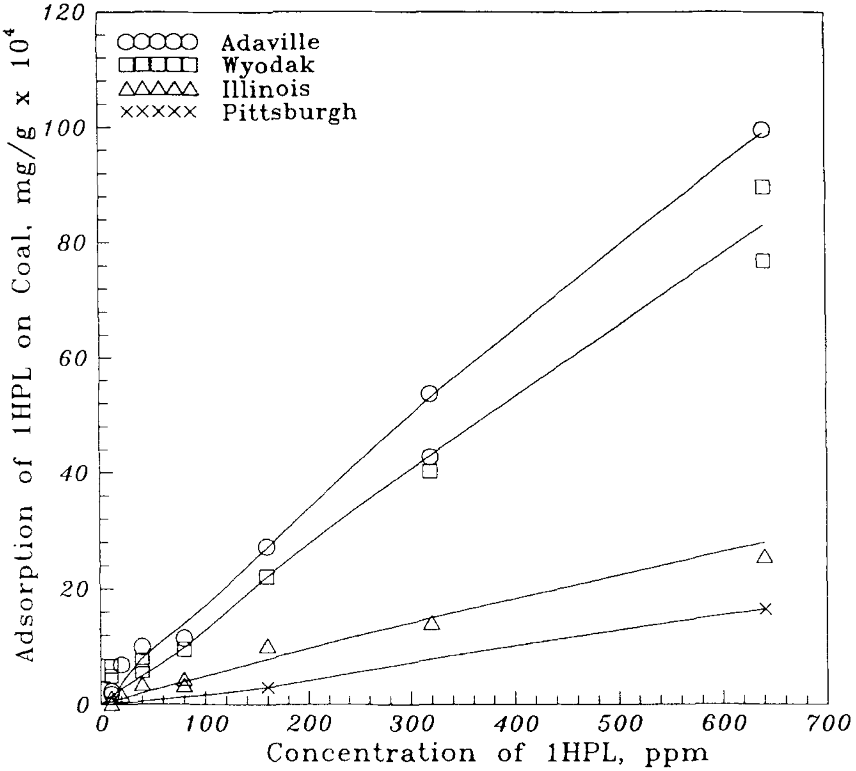

FIGURE 6. Equilibrium adsorption of 1-heptanol (1HPL) on various 0.75-g 60-200 mesh coals in aqueous solutions at room temperature.

Flotation of the coals, equilibrated with aqueous solutions of both 2M1P and 1-heptanol, is plotted against equilibrium adsorption loadings of both 2M1P and 1-heptanol on the coals (see Figures 5 and 8). Flotation of the coals increases with increased adsorption loadings of both 2M1P and 1-heptanol on the coals. These data may indicate that surface properties of coal can be physically modified with higher alcohols in order to enhance separation of mineral matters from organic matters of coal with the flotation method. This

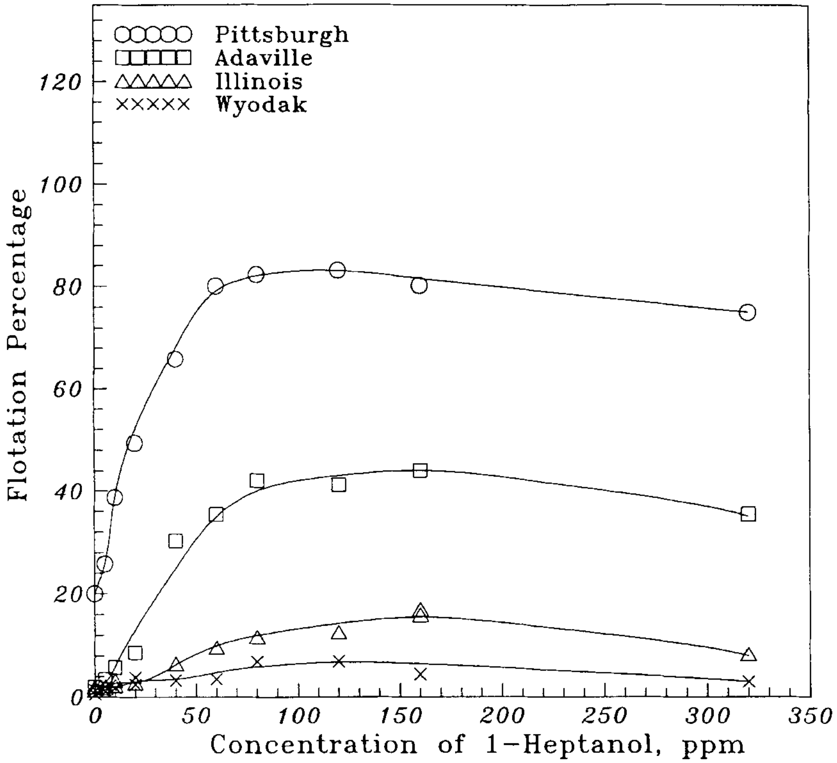

FIGURE 7. Flotation of various coals against concentration of 1-heptanol (1HPL) at room temperature.

observation may suggest that higher alcohols are adsorbed on these coals by the physical association of hydroxyl groups of alcohols with oxygen-containing functional groups of these coals.

The chosen coals were treated with nitrogen and air at 1 atm and 125-225°C for 24 h. Flotation experiments of the treated coals were conducted at room temperature, using distilled water only as a flotation medium.

Flotation of Wyodak coal treated with nitrogen gas is higher than that of untreated coal and increases with treatment temperatures (see Figure 9).

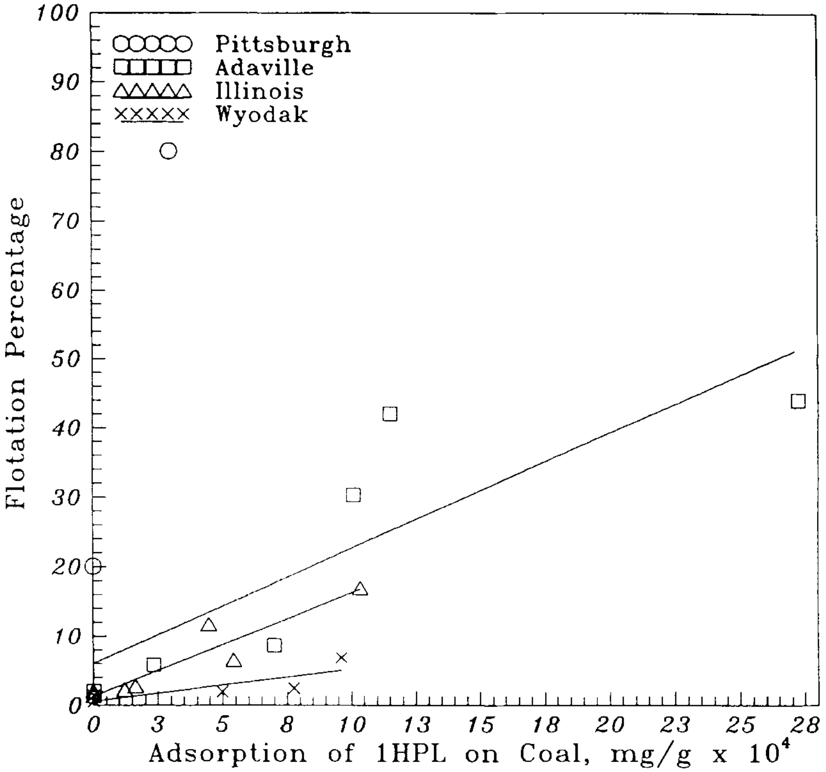

FIGURE 8. Flotation vs. adsorption of 1-heptanol (1HPL) on various coals at room temperature.

Amounts of hydrophilic functional groups of Wyodak coal appear to be reduced with thermal treatments. Thermal decomposition of hydrophilic functional groups increases with treatment temperatures, resulting in enhanced floatability of the coal.

Flotation of Adaville #1 coal treated with nitrogen is higher than that of untreated coal and increases with treatment temperatures (see Figure 10). Amounts of hydrophilic functional groups of Adaville #1 coal also appear to be reduced with thermal treatments. Thermal decomposition of hydrophilic

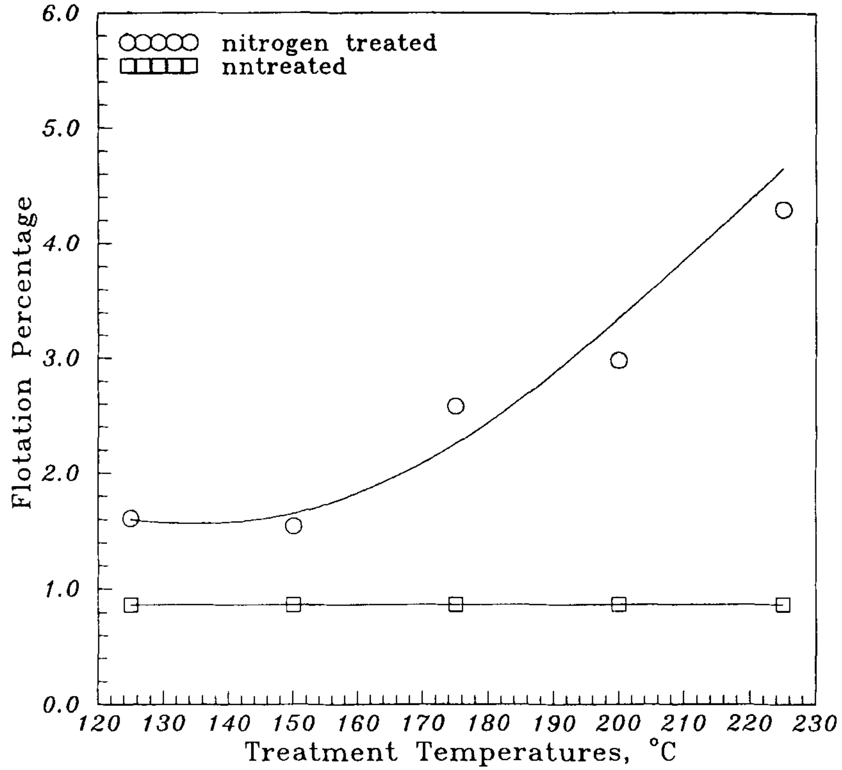

FIGURE 9. Flotation of Wyodak coal at room temperature, using water only as a flotation medium.

functional groups appears to increase with treatment temperatures, resulting in enhanced floatability of the coal.

Flotation of Adaville #1 coal treated with air at 125-150°C is slightly higher than that of untreated coal, whereas flotation of Adaville #1 coal treated with air at 175-225°C is slightly lower than that of untreated coal (see Figure 10). Treating the coal with air over 125 -150°C appears to cause a little higher floatability, resulting in decreased carboxyl groups on the coal surface due to dominant thermal decomposition of carboxyl groups of the coal with nitrogen

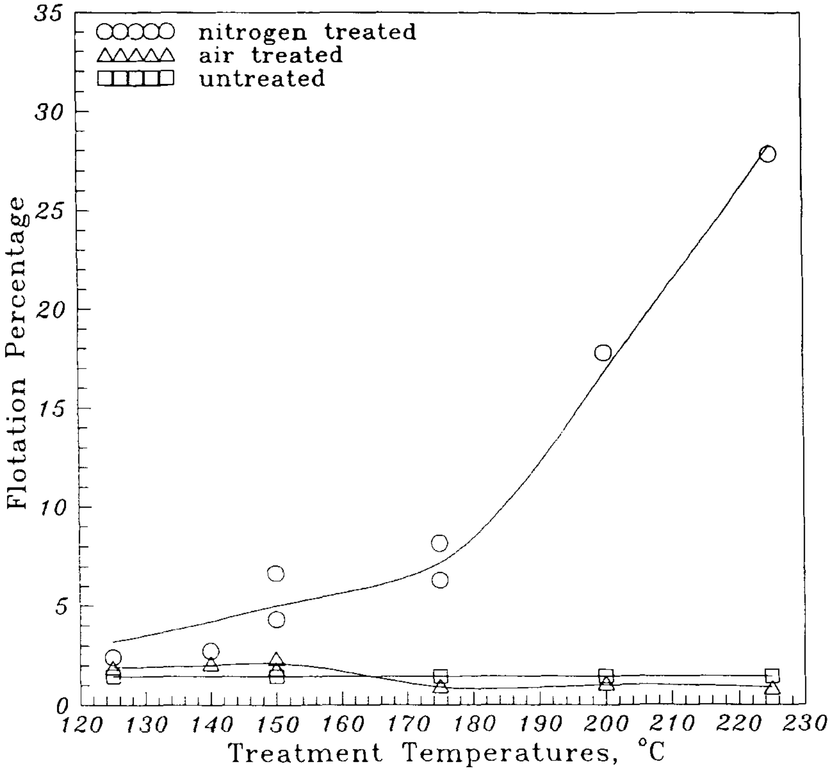

FIGURE 10. Flotation of Adaville #1 coal at room temperature, using water only as a flotation medium.

in air over composition of carboxylic groups with oxygen in air. Treating the coal with air over 175-225°C is adverse to its floatability, resulting in increased carboxylic groups on the coal surface due to dominant composition of carboxylic groups with oxygen in air over thermal decomposition of carboxylic groups with nitrogen in air.

Flotation of Illinois #6 coal treated with nitrogen is higher than that of untreated coal. Flotation of Illinois #6 coal treated with nitrogen at 125-175°C increases with treatment temperatures, whereas flotation of Illinois #6 coal

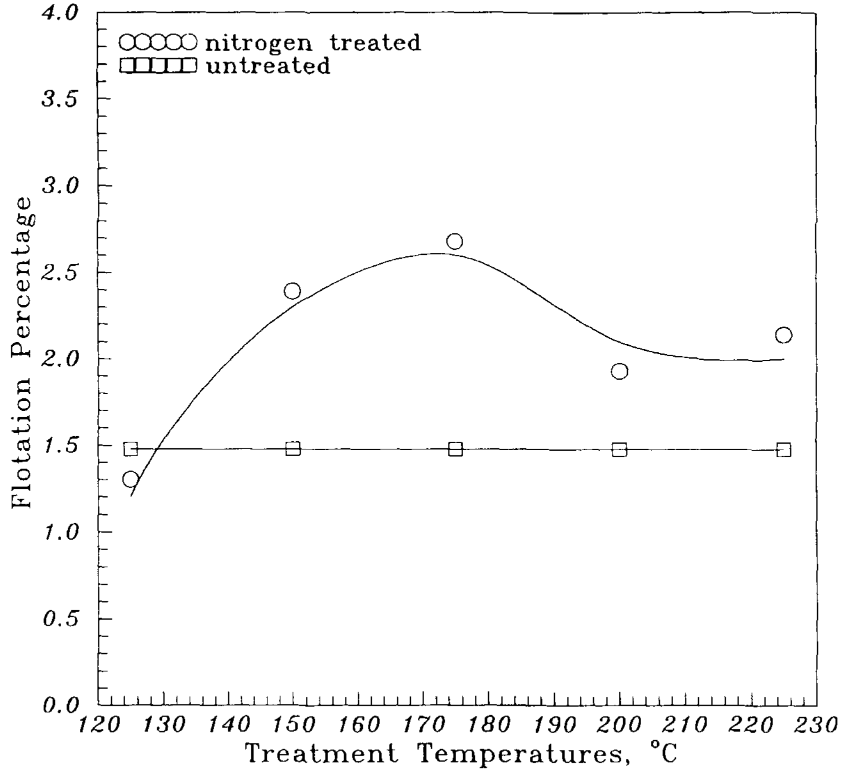

FIGURE 11. Flotation of Illinois #6 coal at room temperature, using water only as a flotation medium.

treated with nitrogen at 175-225°C decreases with treatment temperatures (see Figure 11). Amounts of hydrophilic functional groups of Illinois #6 coal appear to be slightly reduced with thermal treatments in comparison with those of Wyodak coal and Adaville #1 coal. These facts may indicate that Illinois #6 coal appears to contain less carboxylic groups susceptible to themal decomposition than both Wyodak coal and Adaville #1 coal.

Flotation of Pittsburgh #8 coal treated with air is lower than that of untreated coal and decreases with treatment temperatures (see Figure 12).

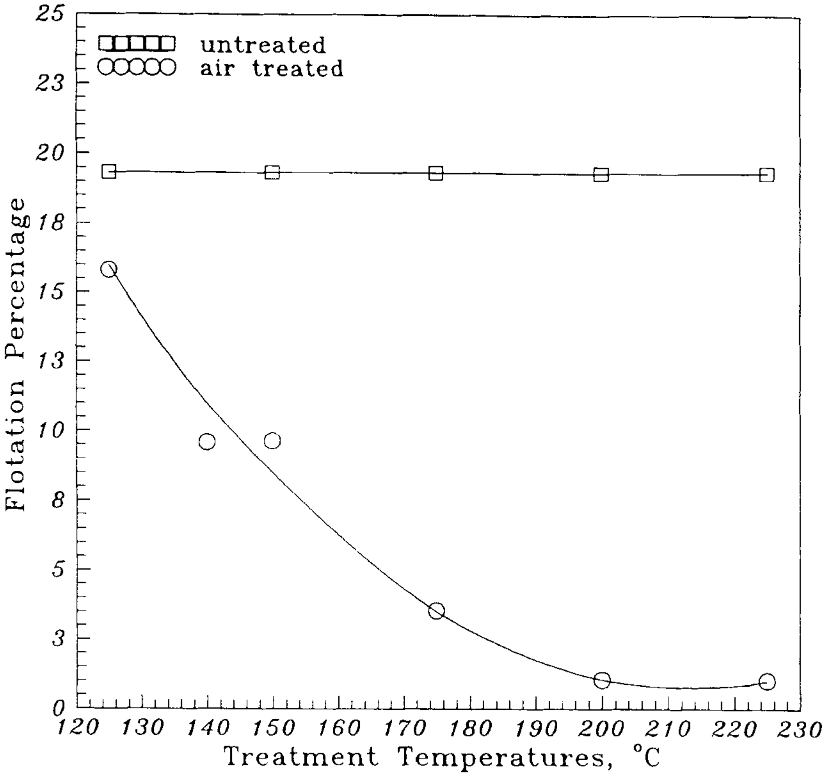

FIGURE 12. Flotation of Pittsburgh #8 coal at room temperature, using water only as a flotation medium.

Oxygen in air appears to increase significantly hydrophilic functional groups (mainly carboxylic group) of Pittsburgh #8 coal with thermal treatments. Untreated Pittsburgh #8 coal appears to contain insignificant amounts of hydrophilic groups vulnerable to thermal decomposition in comparison with the other coals, resulting in monotonically-decreased floatability with increased treatment temperatures.

The above-mentioned experimental results will provide background data for improving chemical technology in coal cleaning/handling processes and optimizing coal cleaning/handling processes by understanding interactions between flotation additives and raw coal as well as treated coal.

CONCLUSIONS

Equilibrium adsorption of aromatic probe compounds such as benzene and toluene on coal increases with decreased coal rank, suggesting that dissolubility of coal in aromatic solvents decreases with increased coal rank. Equilibrium adsorption of aliphatic alcohols such as 2-methyl-1-pentanol and 1-heptanol on coal increases with decreased coal rank, indicating that amounts of hydrophilic functional groups such as carbonyl, carboxyl, methoxyl, and hydroxyl on coal surface increase with decreased coal rank.

Equilibrium adsorption of aromatic probe compounds such as benzene and toluene is much higher than that of aliphatic alcohols on the chosen coals, suggesting that coal structure is more similar to aromatic compounds rather than aliphatic compounds.

Floatability of coal increases with coal rank and adsorption of alcohols on coal. Floatability of both Wyodak coal and Adaville #1 coal treated with nitrogen increases significantly with treatment temperatures in comparison with that of Illinois #6 coal. Floatability of Pittsburgh #8 coal treated with air decreases with increased treatment temperatures, suggesting that oxidation of coal is detrimental to cleaning coal with the flotation method.

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