STUDIES OF EXTRACTION ENHANCEMENT OF COAL THROUGH SUCCESSIVE EXTRACTIONS AND CHEMICAL REACTIONS

By

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DEDICATED TO MY PARENTS

CERTIFICATE

THIS is to certify that the thesis entitled **STUDIES OF EXTRACTION ENHANCEMENT OF COAL THROUGH SUCCESSIVE EXTRACTIONS AND CHEMICAL REACTIONS** by SATYENDRA MISHRA being submitted to the INDIAN INSTITUTE OF TECHNOLOGY DELHI, for the award of the degree of DOCTOR OF PHILOSOPHY is a record of original bonafide research work carried out under my supervision for the submission of this thesis which to my knowledge has reached the requisite standard.

The thesis or any part thereof has not been submitted to any other University or Institution for the award of any degree or diploma.

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ABSTRACT

The present thesis reports the results of an experimental research programme which was aimed to develop some convenient chemical techniques for rendering the coal extractable in common organic solvents under relatively milder conditions at atmospheric pressure, without resorting to high pressure hydrogenation of coal using inorganic catalysts. The thrust was on the use of coal derived chemicals for the pretreatment (chemical reaction) and extraction of coals so that the overall cost of the process is reduced. The objective was to render up to 50% of the coal extractable in quinoline, so that the hydrogen requirement for the subsequent step of hydrogenation of coal extract so obtained can be met through the gasification of the remaining 50% of the coal residue. Bituminous coals from Assam, Godavari, Talcher, Raniganj coal fields and lignite from Neyveli (South Arcot) mines were selected for the present studies.

The studies were performed on the extractive disintegration of coal in coal derived solvents, such as anthracene oil, liquid paraffin and quinoline at their boiling points under atmospheric pressure conditions. More than 50% of the coal (in all) was rendered extractable through successive extraction in these three solvents. Among the different sequences of extraction studied, the anthracene oil - quinoline-liquid paraffin extraction sequence was found to be the best for the maximum extraction of coal (50-68%) through successive extraction. Extraction time for each extraction sequence was optimized. Extractive disintegration of coal with these solvents

resulted in the dehydrogenation of coal. Possible mechanism for the successive sequential extraction has been proposed.

Alkylation reaction has been reported to be a beneficial pretreatment for the liquefaction and gasification of coal. Assam coal was subjected to alkylation reaction using long carbon chain alkenes and alcohols, respectively, in liquid paraffin or in anthracene oil. More than 50% of the coal (in all) was rendered extractable in quinoline as well as in the reaction medium through these alkylation reactions. Alcohols were found to be better alkylating agents than alkenes for rendering the increasing amount of coal extractable. Various reaction conditions for the alkylation reaction of coal in liquid paraffin and in anthracene oil using alkenes or alcohols have been optimised. The extraction yields were higher (50-90%) when anthracene oil was used as a solvent with these alkylating agents than those obtained using liquid paraffin as a solvent with corresponding alkylating agents under the similar conditions (excepting the reaction temperature).

Depolymerization of coal using acid catalyst in phenol has been reported to result in the extraction of more than 80% of the coal in quinoline. In another study it was aimed to reduce the amount of acid catalyst used in this reaction for a desired yield of the extraction of 50% of the coal in quinoline. Pretreating the coal prior to depolymerization reaction or using some promoters in the depolymerization reaction resulted in reducing the amount of acid catalyst in the depolymerization reaction using lesser amounts of reagents. The reaction was found to proceed through demineralization of coal which was probably also responsible for the production of colloidal matter in the coal extracts through depolymerization reaction. A possible mechanism of the depolymerization reaction has been suggested.

The FT-IR, GC-MS and ^{13}CNMR spectral studies of coal extracts obtained through alkylation and depolymerization reactions were also performed. The DTA and TGA studies have been performed to know the physical and devolatilization behaviour of the original coal and of the treated coal products.

These studies may, ultimately, help in developing the cost effective and convenient technology for getting the solvent refined coal at atmospheric pressure and under relatively milder conditions employing cheaper coal derived chemicals and solvents. A convenient two step process for the liquefaction of coal employing solvent extraction under atmospheric pressure conditions has been suggested.

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