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TREATMENT AND REUSE OF COAL CONVERSION WASTEWATERS

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This paper presents a synopsis of recent experimental activities to evaluate processing characteristics of coal conversion wastewaters. Treatment studies have been performed with high-BTU coal gasification process quench waters to assess enhanced removal of organic compounds via powdered activated carbon-activated sludge treatment, and to evaluate a coal gasification wastewater treatment train comprised of sequential processing by ammonia removal, biological oxidation, lime-soda softening, granular activated carbon adsorption, and reverse osmosis. In addition, treatment studies are in progress to evaluate solvent extraction of gasification process wastewater to recover phenolics and to reduce wastewater loading of priority organic pollutants. Biological oxidation of coal gasification wastewater has shown excellent removal efficiencies of major and trace organic contaminants at moderate loadings, addition of powdered activated carbon provides lower effluent COD and color. Gasification process wastewater treated through biological oxidation, lime-soda softening and activated carbon adsorption appears suitable for reuse as cooling tower make-up water. Solvent extraction is an effective means to reduce organic loadings to downstream processing units. In addition, preliminary results have shown that solvent extraction removes chromatographable organic contaminants to low levels.

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INTRODUCTION

Experiments have been performed at Carnegie-Mellon University to characterize coal gasification process wastewaters, to evaluate basic wastewater treatment properties, and to assess wastewater management strategies. The purpose of this paper is to review recent experimental activities in these areas, and to indicate directions for future research.

COAL GASIFICATION WASTEWATER MANAGEMENT

Figure 1 presents a general schematic representation of water streams important in coal gasification process water balances. Major streams include those associated with the boiler and cooling tower systems, process condensates, treatment blowdowns, and slurry/sludge waters. Process influent water streams generally include: water for coal slurry feed, water for direct contact gas cooling or quenching, and water for removal and/or quenching of char, ash, or slag. Process steam requirements include steam to gasifier and make-up steam to CO shift reactor. Process effluents are categorized as slag or ash quench water, raw product gas quench condensate, CO shift condensate, acid gas removal condensate and methanation condensate. The nature and quantities of these process water and effluent streams are highly process specific. The disposition of these streams for particular high BTU coal gasification processes is discussed in Luthy, et al., 1980[1], for the CO_2-Acceptor, Bi-Gas, Hygas, Synthane, and Lurgi processes.

Specific process water treatment and distributional configurations are also strongly dependent on the particular gasification process being considered. Thus various water management schemes exist for different gasification processes. Some aspects of these schemes are well understood and have become generally accepted as necessary in achieving a process water balance. For example, raw makeup water is typically softened and serves as process water, as cooling water, and as supply to the boiler feed water treatment system. In contrast some aspects of high BTU coal gasification process water balance are unique to this industry. This is especially true with respect to treatment and reuse of heavily contaminated phenolic wastewaters. In this case little previous experience is available to detail issues associated with treatment and reuse of these wastewaters; consequently, current research interest is focused on evaluation of specific treatment characteristics for purposes of engineering design and environmental assessment. There is also much interest in evaluating wastewater treatment characteristics in order to achieve a product water of suitable quality for reuse in the gasification process.

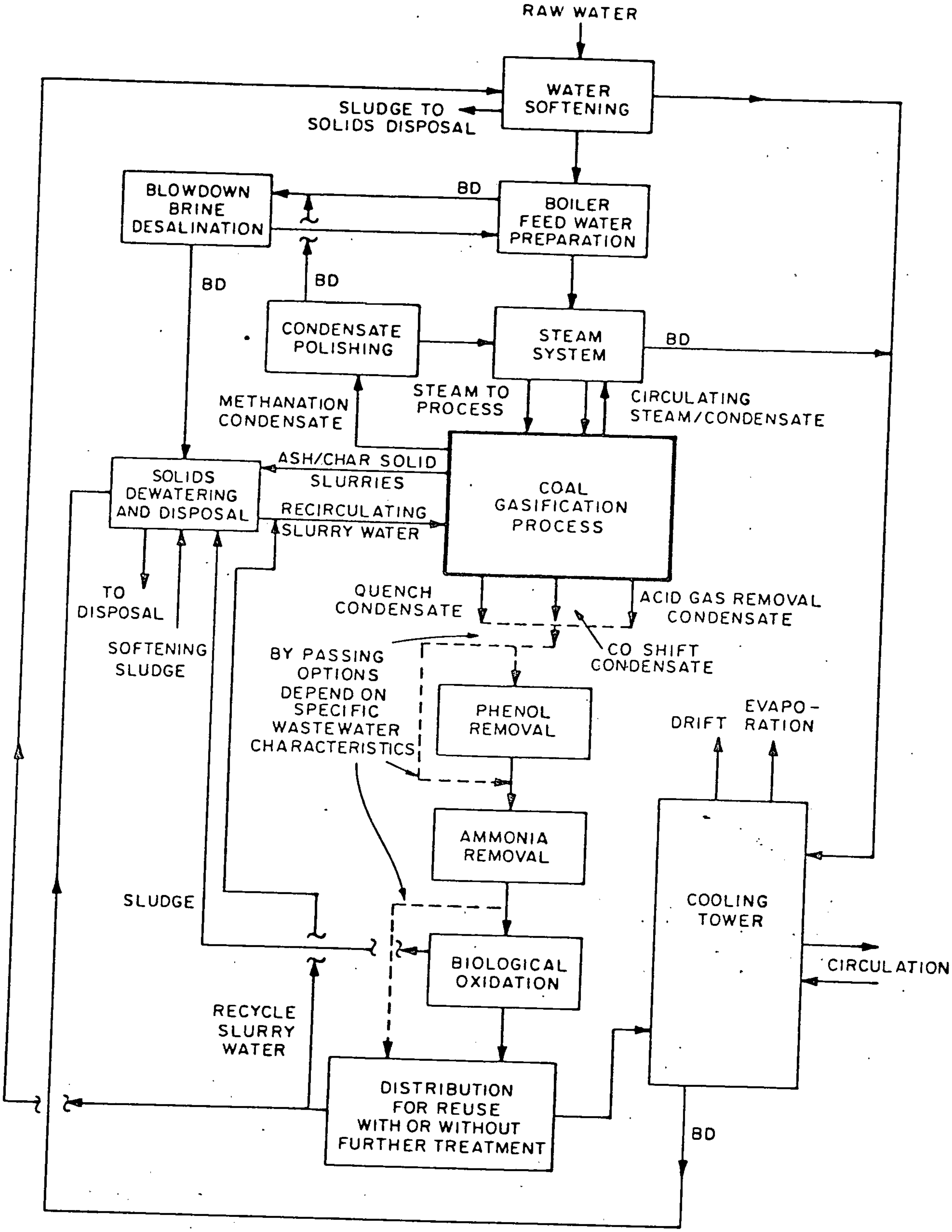

Figure 1

Major water streams in a coal gasification process water balance

Considerations Regarding Water Reuse

Medium and high-BTU coal gasification processes are net consumers of water. The ability to achieve complete water reuse may have a significant impact on the feasibility of a commercial-scale facility, especially for semi-arid western regions and for eastern sites not contiguous or adjacent to large rivers. A general design assumption should hold that all major wastewater streams be considered for reuse, including high organically contaminated streams and saline brines. Dirty water should be cleaned only for reuse and not for discharge to a receiving water; any water suitable for discharge is acceptable for reuse. Returning water to a source is not economic when water must be cleaned to satisfy stringent environmental regulations. Furthermore, treatment for reuse is likely to require less severe processing than treatment for discharge.

Various water management schemes exist for a given gasification process. These depend on the exact nature of the particular waters and on the quality constraints for which waters will be reused. Though specific processes may differ in water management configurations, it is apparent that the cooling tower is the most likely target for wastewater reuse. Treatment for reduction of high ammonia and organic loadings is necessary, while some extent of demineralization and removal of residual organic contaminants will be necessary to achieve a water within quality constraints governing cooling tower makeup. Minimum quality constraints governing acceptable levels of organic contamination in cooling tower make-up are not clearly understood and must be evaluated. Also the fate of toxic hazardous wastewater contaminants during wastewater treatment and during cooling tower operation must be assessed. These factors will ultimately determine the most appropriate treatment scheme to achieve water reuse in a cooling tower.

WASTEWATER CHARACTERISTICS AND SCALABILITY

High-BTU coal gasification processes may be divided into two general classifications with respect to levels of organic contamination in process condensates: 1) those processes which produce little or no phenolics, oils, and tars, and 2) those processes which produce substantial quantities of these materials. Among those processes which produce organic contaminants a further division may be made between those which are significant producers of tars and heavy oils. General data for comparison of coal refinery condensates are presented in Luthy, 1979.[2]

The production of organic contaminants during coal gasification is related to gasifier physical configuration and operating conditions. Processes tending to show little or no organic contamination may be either entrained flow or fluidized bed gasifiers that operate at temperatures greater than approximately 1050°C (1900°F) and produce ash as slag or agglomerates. Examples of such processes are Bi-Gas, Combustion Engineering, Koppers-Totzek, U-Gas, and Westinghouse. Gasifiers having high coal devolatilization temperatures, such as the CO_2-Acceptor process at 830°C (1500°F), also produce a cleaner product gas which in turn yields condensates free of organic contamination (Fillo, 1979[3]). Other important gasifier operating variables which relate to production of organics are gas residence time, coal particle size and heat-up rate, and the extent of gas-solids mixing (Nakles, et al., 1975[4]). Examples of gasification processes which produce effluents with organic contamination are Hygas, Synthane, slagging fixed-bed, Lurgi, and Wellman-Galusha.

It should be recognized that published information on coal gasification process wastewater characterization necessisarily reflects a difference in process scales and use of various coals. Since much of the available data are for analysis of condensates from process development units or pilot plants, it should be expected that any changes anticipated between pilot plant and commercial scale gasifier operating conditions may have significant effects on gasifier effluent production, especially with respect to organic contamination. Thus, scalability of pilot plant data is a major issue in evaluating coal conversion pilot plant effluent composition and distributional trends. Factors to consider may include coal type and pretreatment, coal-to-steam ratio, gasifier geometry and operating parameters, and raw product gas quench system design and operation.

Wastewater treatment experiments performed at Carnegie-Mellon University have utilized process quench waters from the Hygas and slagging fixed-bed coal gasification pilot plants. While these process condensates may not be representative in a quantititative sense of wastewaters which would be expected in a demonstration or commercial scale process, it is anticipated that the majority of organic and inorganic species observed in these effluents may be expected to exist in a commercial facility, though relationships between mass emissions and concentrations may be somewhat different. In as much as the scope of the investigations were to obtain basic information on biological and physico-chemical treatability characteristics of gasification effluents, the pilot plant wastewater samples were envisioned as providing a reasonable matrix of representative contaminants which may be expected in presently conceived commercial facilities.

TREATMENT STUDIES WITH COAL GASIFICATION CONDENSATES

There exists only a limited number of published studies on treatment of organically contaminated coal gasification process wastewaters, especially for the new generation of gasification processes under development. Most of those studies have focused on physico-chemical treatment for reduction of tars, oils, and ammonia prior to biological oxidation, and on basic biological oxidation characteristics of these wastewaters. These data are largely based on experience gained from laboratory bench-scale experimentation.

Experimental biological oxidation studies have been reported for Lurgi coal gasification process effluent (Cooke and Graham, 1965[5]), Synthane (Johnson, et al., 1977[6]; Neufeld, et al., 1978[7]; and Drummond, et al., 1979[8]) and Morgantown Energy Technology Center (METC) pilot coal gasification wastewaters (Sack, 1979[9]), and H-Coal pilot coal liquefaction effluent (Reap, et al., 1977[10]). In addition, biological oxidation studies have been performed with pilot coal gasification process effluents obtained from the Hygas pilot plant operated by the Institute of Gas Technology in Chicago, Illinois (Luthy and Tallon, 1980[11]) and the slagging fixed-bed pilot plant operated by the Grand Forks Energy Technology Center (GFETC) in Grand Forks, North Dakota (Luthy, et al., 1980[12]).

A discussion of performance data and biological oxidation kinetic values for treatment of coal conversion wastewaters is presented in Luthy (1979[2]). A general conclusion from these investigations is that wastewaters processed for removal of ammonia by steam stripping followed by activated sludge treatment for removal of degradable organic matter will show high removal efficiencies for BOD, COD, phenolics and thiocyanate. Nitrification has been demonstrated in several investigations. However, because of the nature of coal gasification process condensates, activated sludge treated wastewater will contain relatively high concentrations of residual organic material. This material is associated with effluent COD and color and is characteristic of oxidation of complex phenolic wastes.

REMOVAL OF TRACE ORGANIC CONTAMINANTS

Less information is available on the trace organic composition of coal gasification wastewaters and removal efficiencies for these compounds during treatment. Singer, et al. (1978) summarizes organic characterization data for coal conversion effluents. Information on removal efficiencies for specific organic compounds from synthetic coal conversion wastewater mixtures is presented in Singer, et al. (1978[13], 1979[14]).

Stamoudis and Luthy (1980[15]) provide results of screening gas chromatography/mass spectrometry analysis of Hygas and GFETC pilot plant wastewater to determine removal efficiencies during biological oxidation. In these investigations wastewater was pretreated by lime addition and air stripping to reduce excess alkalinity and ammonia prior to biological oxidation. The biological reactors were complete-mix, single-stage air activated sludge reactors, with GFETC wastewater being treated at 33% strength and Hygas condensate at 100% strength. General operating parameters and performance characteristics for the biological reactors employed for evaluation of removal efficiencies of organic constituents are summarized in Stamoudis and Luthy (1980[15]). Samples of reacter influent and effluent were prepared for GC/MS analysis by extraction with methylene chloride using generally accepted techniques into acid, base and neutral fractions.

It was found that approximately 99% of influent extractable and chromatographable organic material, on a mass basis, was derivatives of phenol and represented in the acid fraction of the influent samples. Activated sludge processing removed most of the organic constituents, with compounds of the acidic fractions being removed almost completely. High removal efficiencies were also observed for compounds in the basic fraction, with the exception of certain alkylated pyridines. The extent of removal of compounds in the neutral fractions was dependent on chemical structure. Aromatic hydrocarbons containing aliphatic substitutions and certain polynuclear aromatic compounds were only partially removed. A general broad conclusion from this study was biological oxidation provides good to excellent removal for most compounds present in the coal gasification process wastewater.

Followup studies were conducted with GFETC slagging fixed-bed pilot plant wastewater pretreated in the same fashion as above in order to compare removal of organic contaminants by activated sludge and powdered activated carbon (PAC)-activated sludge treatment. Details of the experimental procedures and results are presented in Luthy, et al. (1980[1]).

A high suface area PAC (Amoco PX-21) was selected for use in this study on the basis of results from wastewater batch adsorption isotherm testing. PAC-activated sludge treatment was evaluated at sludge ages of twenty and forty days with PAC mixed liquor equilibrium concentrations of 0, 500, 1500, and 5000 mg/l. The reactors were operated for an appropriate balance period to achieve steady state operation.

Activated sludge treatment with no addition of PAC showed excellent removal of phenolics and BOD. Phenolics were reduced to less than 1 mg/l from influent values of 1300-1500 mg/l; BOD was reduced to about 30 mg/l from influent concentration of 3600-3800 mg/l. COD removal efficiencies were 85% and 88% at removal rates of 0.37 and 0.24 mg COD removes/mg MLVSS-day at sludge ages of twenty and forty days, respectively.

PAC-activated sludge treatment gave significantly lower effluent COD and color with increasing equilibrium carbon concentrations. In addition, somewhat lower effluent concentrations of BOD, phenolics, ammonia, organic-nitrogen, and thiocyanate were achieved by PAC-activated sludge treatment compared to activated sludge treatment. PAC-activated sludge treatment reduced foaming problems and gave a sludge with good settling properties. Effluent characteristics were not significantly different for PAC-activated sludge treatment at a sludge age of twenty and forty days. In general, PAC-activated sludge treatment in this study gave as good or better effluent characteristics than previously reported results with other industrial wastes. A highly nitrified effluent was produced by PAC-activated sludge treatment at a sludge age of forty days. This effluent appears suitable for reuse as cooling tower make-up water with respect to macro-organic contaminants.

Samples of biological reactor effluent with sludge age of forty days and mixed liquor PAC concentrations of 0, 500, 1500, and 5000 mg/l were screened for base and neutral fraction organic compounds. Base and neutral fraction capillary column chromatograms of all four reactors were very similar. Characterization of sixteen compounds, representing some of those which were found not to be completely removed in the previous GC/MS study with slagging fixed-bed wastewater, gave similar GC flame ionization detector responses in effluent samples for all four reactors with concentration levels of these compounds in the range of several mg/l. These results confirmed that biological oxidation of coal gasification wastewaters removes organic contaminants to low levels, however PAC-activated sludge treatment does not necessarily provide significantly lower effluent concentrations of certain trace organic compounds under conditions in which the biological oxidation process has been optimized. The PAC results can be explained in part on competition adsorption between very low concentration of base and neutral fraction compounds and very high concentration of oxidized and/or polymeric substances resulting from biological treatment of phenolic wastes. These later substances are similar to humic materials and are associated with residual effluent COD and color. These substances are removed significantly by PAC-activated sludge treatment, and they likely compete with trace organic contaminants for adsorption on the powdered activated carbon.

EVALUATION OF A COAL GASIFICATION WASTEWATER TREATMENT TRAIN

A sample of Hygas pilot plant Run 79 coal gasification quench condensate has been processed through sequential wastewater treatment unit operations to evaluate treatment technology to achieve wastewater reuse. The unit operations investigated in this study are shown in Figure 2 and include: ammonia removal, biological oxidation, lime-soda softening, activated carbon adsorption, and reverse osmosis.

The raw wastewater contained approximately 0.86 meqv/l of alkalinity and 0.94 meqv/l of ammonia at pH of 7.7. These results plus batch steam stripping tests showed that approximately 97% of the ammonia can be liberated in one unit operation without chemical addition. Removal of the remaining fraction of ammonia will require addition of lime or caustic. If lime is used, this will result in a significant increase in wastewater hardness (>1000 mg/l as $CaCO_3$). In this study, steam stripping was simulated by liming to precipitate alkalinity and air stripping to remove ammonia. The residual hardness in stripped wastewater was in the same range regardless if free- and fixed-leg steam stripping or liming and air stripping were used for ammonia removal.

Biological oxidation at a COD removal rate of 0.16 mg COD removed/mg MLVSS-day gave 90% reduction in COD from an influent value of 6900 mg/l, and 99% reduction in BOD from an influent value of 3500 mg/l. There was also 96% removal of thiocyanate and reduction of phenolics to 0.7 mg/l. Biologically treated wastewater contained about 30 mg/l BOD, 700 mg/l COD, and 1200 mg/l hardness (as $CaCO_3$). It was judged that if biologically treated wastewater were to be used as make-up to a cooling tower, that the COD was sufficiently high to promote potentially significant biological activity, and that calcium and sulfate levels could lead to scaling and fouling problems. Therefore, removal of calcium hardness was evaluated by lime-soda softening,

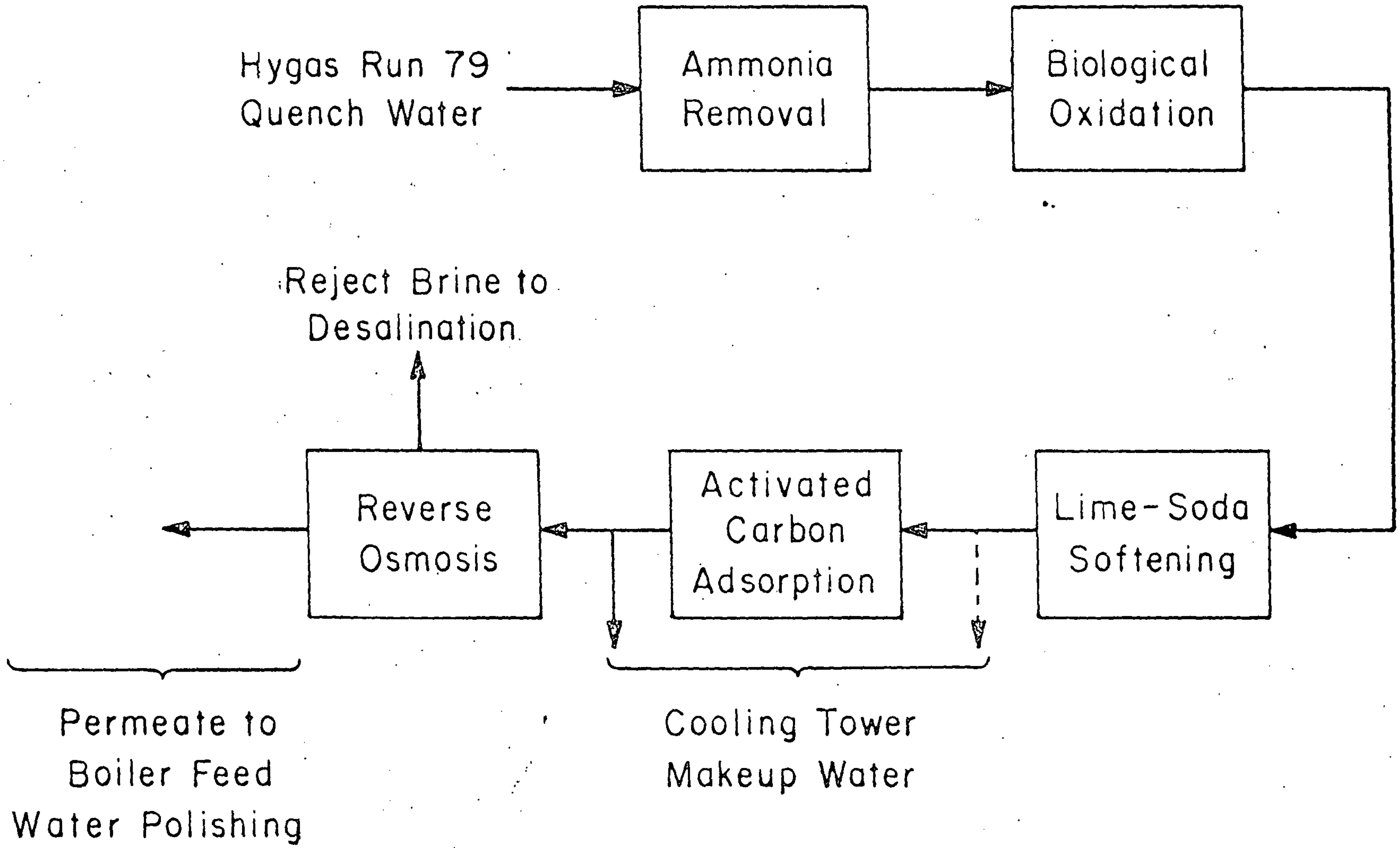

Figure 2

Bench Scale Treatment Train to Evaluate Processing Characteristics of Hygas Process Quench Condensate and removal of COD was assessed by granular activated carbon treatment of softened wastewater.

Most of the calcium hardness in biological reactor effluent existed as non-carbonate hardness owing to the consumption of alkalinity during biological oxidation. Thus, lime-soda softening required proportionally more soda than lime. This resulted in the replacement of residual wastewater equivalents of hardness by equivalents of sodium. Lime-soda softening reduced wastewater hardness to practical limits (30-40 mg/l as $CaCO_3$). These tests also indicated that flocculation and/or filtration would be necessary to clarify sludge formed by the softening operation. Granular activated carbon adsorption column testing of softened biological effluent was conducted at pH of 7, a contact time of seventeen minutes, and a loading of about 1.2 gpm/ft^2. These tests showed that approximately 80% of COD and 95% of residual color could be removed by carbon adsorption.

Hygas wastewater processed by ammonia removal, biological oxidation, lime-soda softening, and activated carbon adsorption was judged to be of sufficient quality for reuse as cooling tower make-up water. At this time it is not possible to predict the degree of cooling tower biological activity which may be induced by residual COD of about 100 mg/l in carbon treated effluent, although it is suspected that a biocidal program could control this problem.

Reverse osmosis experiments were conducted with granular activated carbon treated wastewater. Reverse osmosis treatment with a hollow fiber polyamide membrane produced a clear colorless product, with a TDS level comparable to tap water. Low levels of organic contaminants (COD = 20 mg/l) did permeate the membrane. It is believe that these compounds were low molecular weight, and that they permeated the membrane owing to preferential sorption at the membrane-solution interface. Product water from reverse osmosis treatment is suitable for reuse as make-up to a boiler feed water polishing facility.

Reverse osmosis membrane fouling was not observed in this study under operation at 75 percent conversion. Addition of a polyphosphate inhibitor is thought to have been at least partially responsible for this. A decline in membrane flux did occur, but this was primarily a result of membrane compaction. Comparison of polyamide and cellulose triacetate hollow fiber membranes showed that the polyamide membrane provided a higher quality product water while the cellulose triacetate membrane provided higher flux rates.

This investigation showed that a possible treatment scheme for reuse of phenolic coal gasification effluents may include provisions for ammonia stripping, biological oxidation, softening, and activated carbon adsorption. These unit processes will provide a water with sufficient quality for reuse as cooling tower make-up water. Further study is required to assess the possibility of excessive biological activity and/or emissions of trace compounds to the environment as a result of wastewater reuse in cooling towers. Resolution of this problem may depend on large pilot cooling tower studies and on operational experience gathered at demonstration plants.

Reverse osmosis appears to be an attractive technique to remove wastewater dissolved solids. If reverse osmosis is employed in treatment system design, the resulting product water will be of sufficient quality to be used as a boiler feedwater source. However, further study needs to be undertaken to determine the extent of membrane fouling that could possibly occur under long term steady state operation. It is probably best to evaluate reverse osmosis treatment units at the pilot scale once demonstration plants have been built.

EVALUATION OF A PROPOSED TREATMENT TRAIN FOR A DEMONSTRATION PLANT

Figure 3 shows a simplified schematic of a proposed wastewater treatment system for a slagging Lurgi process to gasify Illinois No. 6 bituminous coal (Continental Oil Company, 1979[16]). Wastewater treatment at this proposed facility handles streams discharging to an oily water sewer, Rectisol process blowdown, solvent extracted wastewater from ammonia recovery, and sanitary wastewater. As shown in Figure 3, the treatment train for wastewater from ammonia recovery passes to an equalization basin and then to a dissolved air flotation unit. Wastewater is then treated biologically in an extended aeration basin of three days hydraulic detention time. Effluent from the biological reactors is clarified, processed through polishing filters, and then pumped through granular activated carbon columns for removal of residual organics. Wastewater from the activated carbon unit is pumped to the utilities cooling tower.

The utilities cooling tower supplies cooling water to equipment having ordinary or carbon steel metallurgy. Makeup to the utilities cooling tower is obtained from various sources of which blowdown from the process cooling tower comprises the largest portion of the total. Makeup from wastewater treatment comprises about 17% of the total demand. The plant is designed for zero discharge of wastewater. The key units for this are multi-stage and Carver-Greenfield evaporators. The multi-stage evaporator concentrates an approximate one percent feed to an approximate 30 weight percent salt solution. The condensate is recovered in the utility cooling tower and the salt solution is concentrated to an approximate 60 weight percent aqueous slurry. The concentrated salt mixture is chemically fixed and trucked to a landfill. Continental Oil Company recommended that semi-commercial evaporators be constructed and evaluated prior to constructing large units because no commercial experience exists with wastewater from a gasification facility, and there may be problems with scaling and foaming.

Figure 4 shows a schematic representation of experiments in progress to evaluate essential features of a wastewater treatment train of the

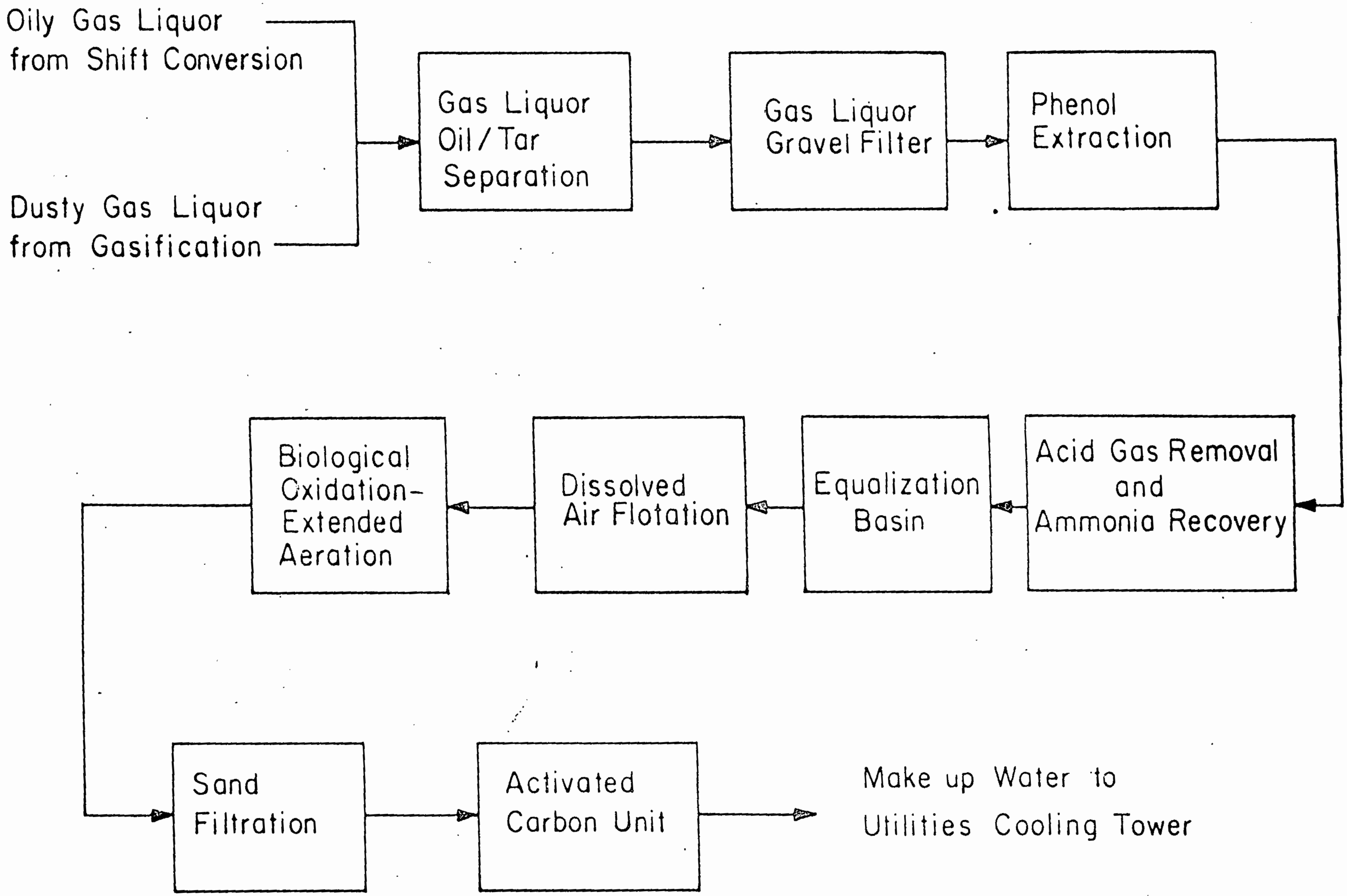

Figure 3

Proposed wastewater management scheme for a Lurgi plant gasifying Illinois No. 6 bituminous coal (Continental Oil Company, 1979)

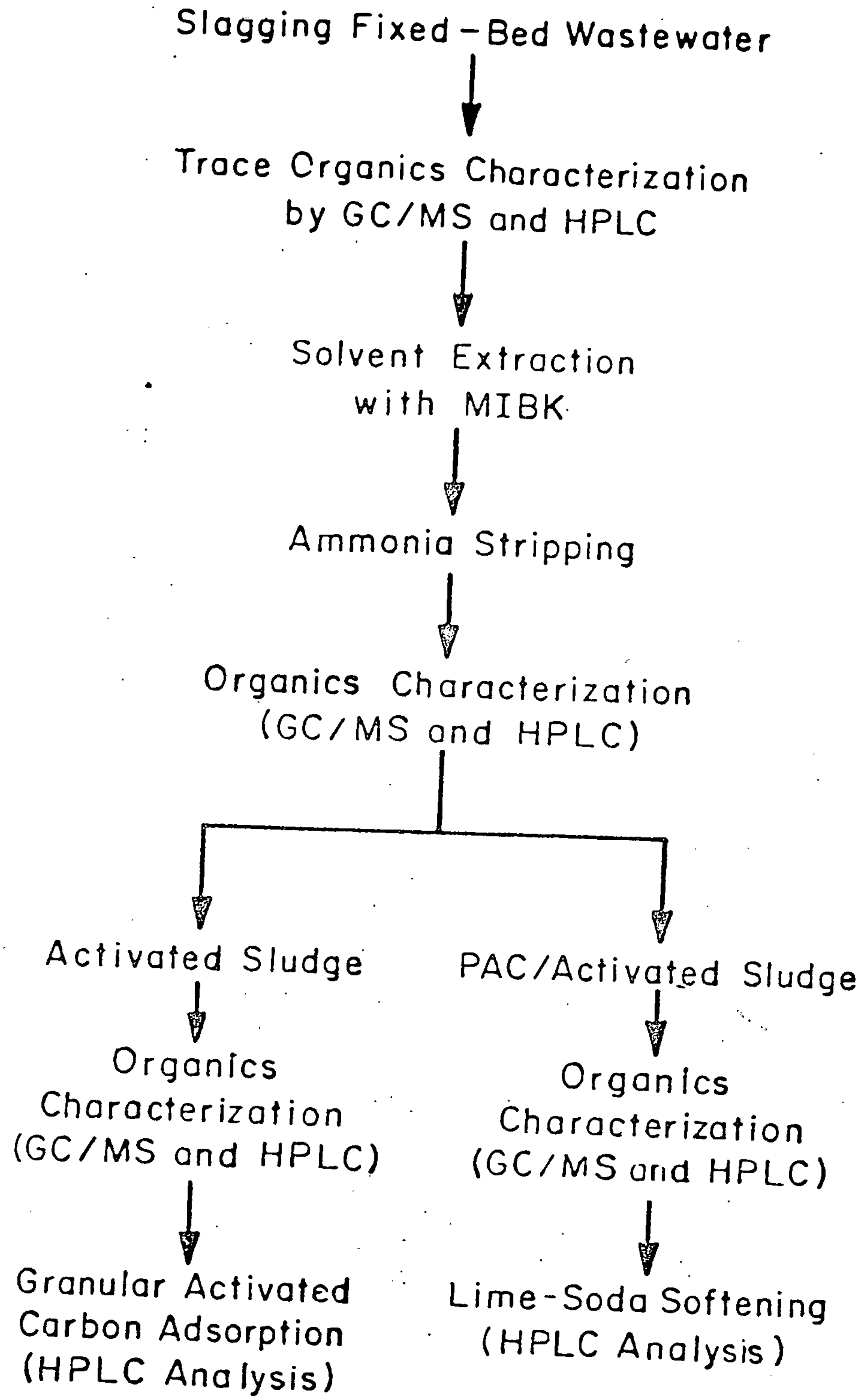

Figure 4

Experiments in progress to evaluate essential features of a coal gasification wastewater treatment train.

type discussed above. This study utilizes GFETC slagging fixed-bed lignite wastewater without dilution. Wastewater is processed through solvent extraction, steam stripping, and biological oxidation with and without PAC addition. Effluent from biological oxidation with no PAC is treated by granular activated carbon adsorption, while effluent from the PAC-activated sludge reactor is evaluated for lime-soda softening characteristics. High pressure liquid chromatographic analyses are being performed after each treatment step to assess removal of polycyclic aromatic hydrocarbons. Screening GC/MS analyses are being conducted on raw, solvent extracted-ammonia stripped, and activated sludge and PAC-activated sludge effluent to characterize removal efficiencies for trace organic contaminants. At this writing, experiments have been completed through biological oxidation. Gas chromatography and GC/MS scans have been made for raw, solvent extracted-ammonia stripped, and PAC-actived sludge effluent. A report on the results of this investigation should be available for distribution later this year.

Several representative solvents were screened for use in the solvent extraction step. As a result of this analysis methylisobutyl ketone was selected for use owing to its measured high distribution coefficient for phenolics. Wastewater was processed through five sequential extraction steps at a solvent-to-liquid ratio of 1:15. This reduced phenolics from 5500 mg/l to about 5 mg/l. Concomitant with phenolics removal there was 88% reduction of COD (32,000 to 3900 mg/l) and 89% removal of BOD (26,000 to 2900 mg/l). Preliminary evaluation of GC/MS data suggests that there is on the order of 99%+ removal for most organic compounds through solvent extraction and ammonia stripping.

It has been demonstrated that solvent extracted wastewater can be processed by either activated sludge and PAC-activated sludge treatment without the need for dilution. Additionally, solvent extracted wastewater does not show tendency to foam excessively as observed in previous investigations. Effluent BOD values were in the range of 30 mg/l for both activated sludge and PAC-activated sludge treatment. PAC treatment showed generally better removal efficiency for TOC, COD, ammonia-nitrogen, organic-nitrogen, SCN^-, and color. Initial assessment of GC/MS scans of extracts from activated sludge and PAC-activated sludge treated wastewater indicates that organics are reduced to extremely low levels, generally less than several micrograms per liter.

This work has shown that solvent extraction offers several distinct wastewater processing advantages. Aside from recovering phenolics for use as a fuel or chemical commodity, there is achieved a marked reduction of trace organic compounds. If the extract is to be used for fuel, then there is the possibility of combusting toxic/hazardous organic compounds to thermal extinction. Solvent extraction reduces organic loading to a biological oxidation facility, and it may also serve as a physico-chemical treatment step to moderate shock loadings of organics. Solvent extracted coal gasification process wastewater is easier to treat biologically than wastewater which would otherwise contain much higher levels of organics.

FUTURE WORK

It is planned to continue these investigations in order to understand removal efficiencies and fates of trace organic compounds during treatment of wastewaters derived from production of synthetic fuels. Preparations are being made to perform experiments with slagging fixed-bed wastewater generated from conversion bituminous coal. Data gained from this study will be used to develop a model for predicting the fates of various trace organic contaminants during treatment with special emphasis on modeling removal of trace organics during solvent extraction. It is also proposed to conduct analogous investigations with oil shale and tar sand condensates where the objective of these studies would be to characterize and evaluate removal of organic compounds via proposed treatment trains for demonstration facilities.

ACKNOWLEDGEMENTS

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