BIODESULF™, A Novel Biological Technology for the Removal of H_2S From Sour Natural Gas

Kailash C. Srivastava (bioproce@arctech.com; 703-222-0280)
Joseph J. Stashick (analytic@arctech.com; 703-222-0280)
Priscilla E. Johnston (bioproce@arctech.com; 703-222-0280)
Nand K. Kaushik (actosol@arctech.com; 703-222-0280)
ARCTECH, Inc.
14100 Park Meadow Drive, Suite 210
Chantilly, VA 20151

Introduction[1]

The state-of-the-art technologies for the removal of sulfur compounds from Sour Natural Gas SNG) are not cost-effective when scaled down to approximately 2-5 MMSCFD. At the same time, the SNG Production is increasing @ 3-6 TCF/Yr and ~78 TCF potential reserves are also sour. Assuming only 3% treatment of this potential SNG market is for small volume processing, the potential U.S. Market is worth $0.14 to $0.28 billion. Therefore, the Gas Processing Industry is seeking novel, cost-effective, environmentally compatible and operator friendly technologies applicable to the small volume producers in the range of $\leq$ 1 MMSCFD to ~ 5 MMSCFD.

A novel biological process, BIODESULF™ (patent pending), developed at ARCTECH removes H_2S and other sulfur contaminants that make the Natural Gas Sour. The removal is accomplished by utilizing an adapted mixed microbial culture (consortium). A variety of anaerobic microbial consortia from ARCTECH's Microbial Culture Collection were grown and tested for removal of H_2S. One of these consortia, termed SS-II was found to be particularly effective. Utilizing the SS-II consortium, a process has been developed on a laboratory-scale to remove sulfur species from Sour Natural Gas at well head production pressures and temperatures. The process has been independently evaluated[15] and found to be promising in effectively removing H_2S and other sulfur species cost effectively.

Objective

In recent years several microbiological processes have been investigated that utilize chemoautotrophic bacteria[5,10,16&17], belonging to Genus *Thiobacillus*[2]. In the direct treat biological processes such as BIODESULF™, the bacteria of the Genus *Thiobacillus* oxidize the sulfide species by using them as a source of energy in the presence of CO_2 or HCO_3^{-1} as the carbon

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source, and NH_4^{+1} or NO_3^{-1} as the source of nitrogen[2,] according to the following chemical equation:

$$0.422H_2S + 0.422HS^{-1} + NO_3^{-1} + 0.346\ CO_2 + 0.0685\ HCO_3^{-1} + 0.0865\ NH$$
$$0.844\ SO_4^{-2} + 0.5\ N_2 + 0.288\ H^+ + 0.4025\ H_2O + 0.0865\ C_5H_7O$$

In this reaction, the H_2S and other reduced sulfur species (HS^-) serve as the source of energy, while CO_2 from the natural gas and HCO_3^{-1} from the nutrient solution are the source of carbon. The NH_4^+ or NO_3^{-1} also present in the nutrient solution for the growth of bacteria serve as the source of nitrogen. Since the BIODESULF™ process is under anaerobic conditions, the NO_3^{-1} also serves as alternate electron donor in the absence of molecular O_2.

Literature shows that in contrast to a homogeneous culture consisting of only one bacterial species, a mixed culture, such as a bacterial or microbial consortium consisting of different morpho- and physiotypes of bacteria/microorganisms, brings about 75% higher sulfide oxidation than that catalyzed by a chemical agent. The important condition, however, is that the sulfide concentration should be ≤ 3 mm and reaction conditions should be favorable for the microbial metabolism[Cf.17]. With these considerations, the objectives for the development of the BIODESULF™ Process (patent pending) were to develop a biological process for the sweetening of SNG which:

- meets pipeline specifications (`≤ 4 ppm H_2S and regulatory limits for other sulfur species, such as COS, CS_2, CH_3SH, $[CH_3]_2SH$, C_2H_5SH, and CO_2),
- achieves specific sulfur removal rates of at least 0.1 lb/ft^3h^{-1}
- is applicable for small scale operations (~0.2 to 2 TPD of sulfur), and
- reduces costs for desouring the SNG in comparison to existing state-of-the-art technologies.

Therefore, the following process parameters are being investigated:

- reaction rates,
- gas delivery system (mass transfer),
- pressure, pH and temperature,
- nutrition cost and availability,
- biomass loading effects,

- handling considerations, major one being the viscosity of the cell suspension,

- amounts of by-products of the metabolism such as sludge produced and disposal of the sludge,

- shear stress on the microorganisms during the process,

- reactor systems and materials of construction for the reactors, and

- overall process costs.

The specific objectives for this project under the DOE Grant are:

- conduct additional process research to optimize certain critical parameters governing the process,

- to construct and operate a pre-pilot scale (50 liters) biologically-based process for sweetening SNG, and

- design, construct and operate a pre-pilot unit.

Approach

The information presented in this paper is the result of a careful systematic approach toward developing a process from the test tube to the process design on a pre-pilot scale. The research was conducted in three phases as described below:

- Laboratory Scale Research for the Proof of Concept,

- Bench scale Research at Atmospheric Pressure, and

- Process Research at Pressures higher than 1 atmosphere.

Laboratory Scale Research For The Proof Of Concept Preliminary results from the experiments conducted on the laboratory scale in microbioreactors showed that:

- In contrast to other microorganisms and microbial consortia tested, the SSII consortium showed highest growth in presence of H_2S. Moreover, SSII microbial consortium pregrown in presence of thiosulfate showed higher removal rates of H_2S than those that were pregrown in presence of H_2S.

- Preliminary data established the technical feasibility of biological oxidation of H_2S from 10,000 ppmV to $\leq$ 4 ppmV under anaerobic conditions.

- The data from these experiments also indicate that the SSII microbial consortium can remove the H_2S as well as other components of the SNG, namely: COS, and mercaptans (dimethyl, ethyl, and methyl).

- Next the removal of H_2S was tested at higher flow rates and in continuous operation for at least 96 hours, and these data indicate that the SSII consortium has the potential to adapt to the conditions that prevail in the SNG industry and the process can be scaled-up.

Bench Scale Research At Atmospheric Pressure Further experiments at higher flow rates of the simulated sour natural gas (SSNG) in the 14-L reactor established that the H_2S removal rates obtained by the SSII consortium are almost 4 times as high as those obtained by the scavenging technologies. At atmospheric pressure, and up to a flow rate of approximately 3L/h, the H_2S removal by the SSII microbial consortium was ~ 0.4 $lb/ft^3\ h^{-1}$.

Experiments conducted in the 14-L bioreactor also established the following bench scale process parameters for the biological removal of the sulfur contaminants from the SSNG:

- a source of organic nitrogen is required for the microbial metabolism,

- thiosulfate is required only to initiate the removal of the target sulfur species form the SSNG,

- methane is not consumed, but CO_2 is reduced from ~10% to ~5%

- microbial activity takes place in the temperature range of 86°F to 104°F (30°C to 37°C), and in the pH range of 7.5-8.

- two orders of magnitude (300 min to 3 min) reduction in contact time was achieved at 10 fold higher SSNG flow rates)

- the microbial process takes place at 1,000 psi.

Process Research At Pressures Higher Than 1 Atmosphere This work focused on investigating several process parameters of interest to take the biodesulfurization of SNG to the next stage of pre-pilot development. This is also the subject of ongoing research under the current grant from the Federal Energy Technology Center, Morgantown.

Project Description/Technology

The data obtained thus far indicate that this biological process will be ideally suited to the removal of H_2S from marginal gas from a well head. The process may also be useful for the sweetening of the fuel gas feed for off-shore turbo-generators.

Data developed thus far, while promising, was generated at a bench-scale typical of the laboratory environment. Now that the concept has been proven in the laboratory, the next logical step is to scale up the process to a larger scale and determine as well as establish key engineering scaling-up factors.

The specific milestones for testing of this technology are:

- successful development of a pre-pilot reactor,
- installation and operation of the 50-L pre-pilot reactor demonstrating the mitigation of sulfur species common in the SNG,
- successful demonstration of the operation of a pilot scale reactor treating the SNG to obtain pipe line quality gas meeting the regulatory specifications.

The major project objective is to conduct studies on a pre-pilot scale (50 liters). However, there are certain process parameters that still have not been defined. The proposed project under the Grant received from METC consists of three separate tasks:

Task 1 - This task consists of additional experimental studies to evaluate the process parameters that still remain to be definitively defined. These studies will consist of batch and continuous reactor studies and are described in more detail in the subsequent sections.

Task 2 - Continuous testing in 2-L Parr Pressure reactor using the optimum operating parameters evaluated in Task 1, and previous studies.

Task 3 - Prepilot testing with 50-L reactor. Using the optimum process parameters determined under Tasks 1 and 2, the process will be scaled up to a pre-pilot unit (50-L), and operated.

All the work described in this project is being performed in tandem. Each run is being repeated for a total of three (3) times in order to obtain enough data for the statistical analysis of the results. H_2S, COS, CS_2, and mercaptans (dimethyl, ethyl and methyl) will be analyzed by gas chromatography using FPD. The sulfur species (S^{-2}, SO_3^{-2}, SO_4^{-2}, and $S_2O_3^{-2}$ formed during the reaction are being evaluated by HPLC. The experimental set-up used is presented in Figure 1.

Results

The results form the work which focused on investigating several process parameters of interest to take the biodesulfurization of SNG to the next stage of pre-pilot development are discussed in the following paragraphs.

Reduction In Nutrient Cost A number of economical sources of organic nitrogen were examined. Among those examined were the Sheftone line of products from the Sheffield Co., Cargill 200/20, and chicken manures from at least two different sources and at 4 different stages of the maturity of the chicken manure. The experiments show that the commercially available Cargill 200/20 not only is most economical, but also gives highest removal rate as represented by the data presented in Figure 2. Nevertheless, additional investigations are still continuing to identify a source of organic nitrogen that is more cost-effective than this product, but can further enhance the reaction rates.

Figure 1: Experimental Set-Up for Semi Continuous or Batch Reactions at

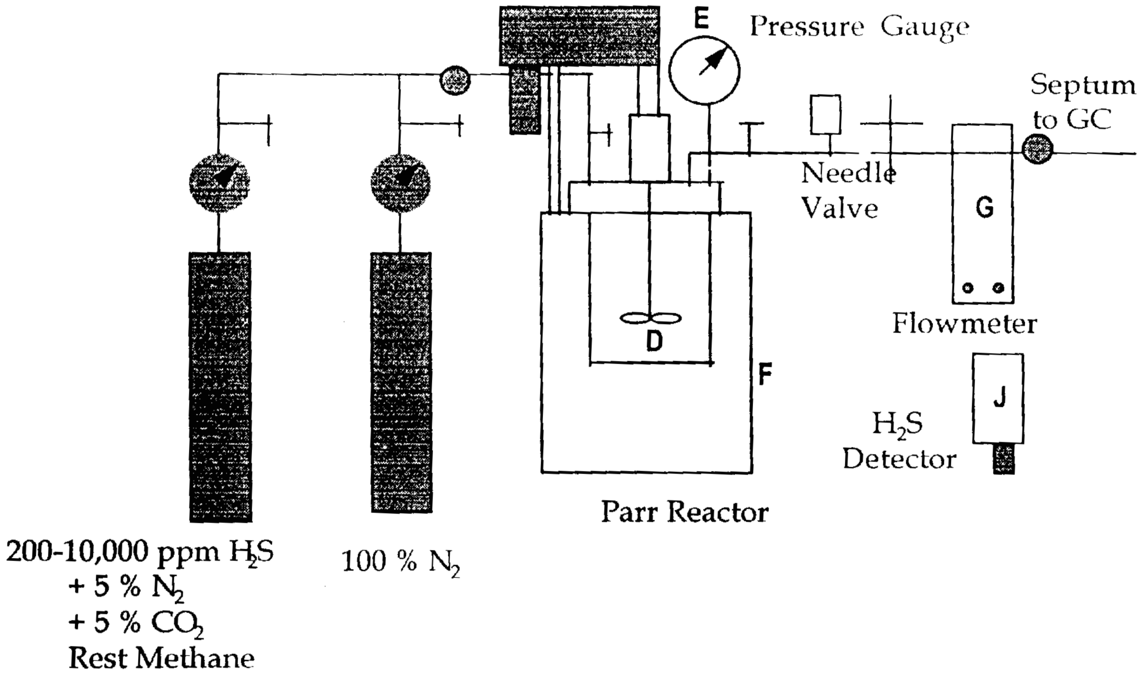
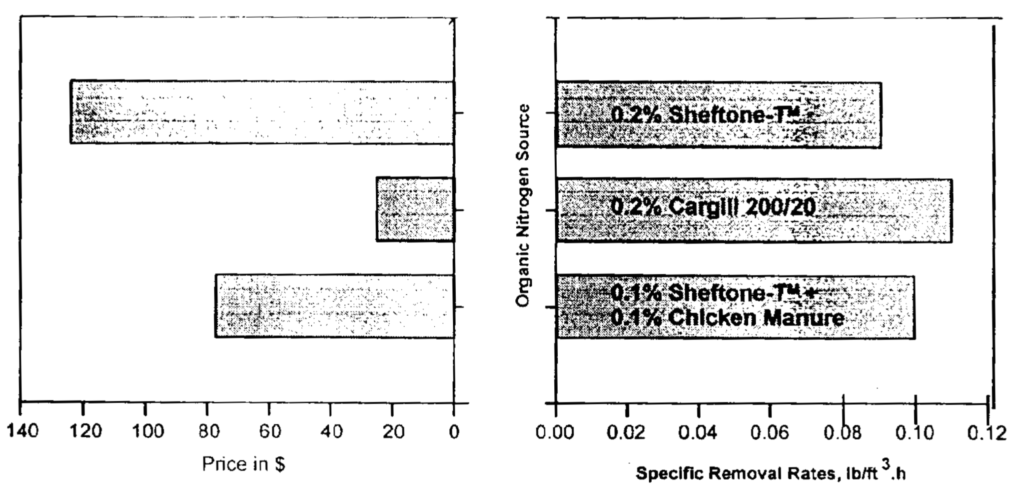


Fig.2: The Cost of Nutrient Medium is Significantly Reduced with Cargill 200/20 Without Compromising H_2S Removal Rates

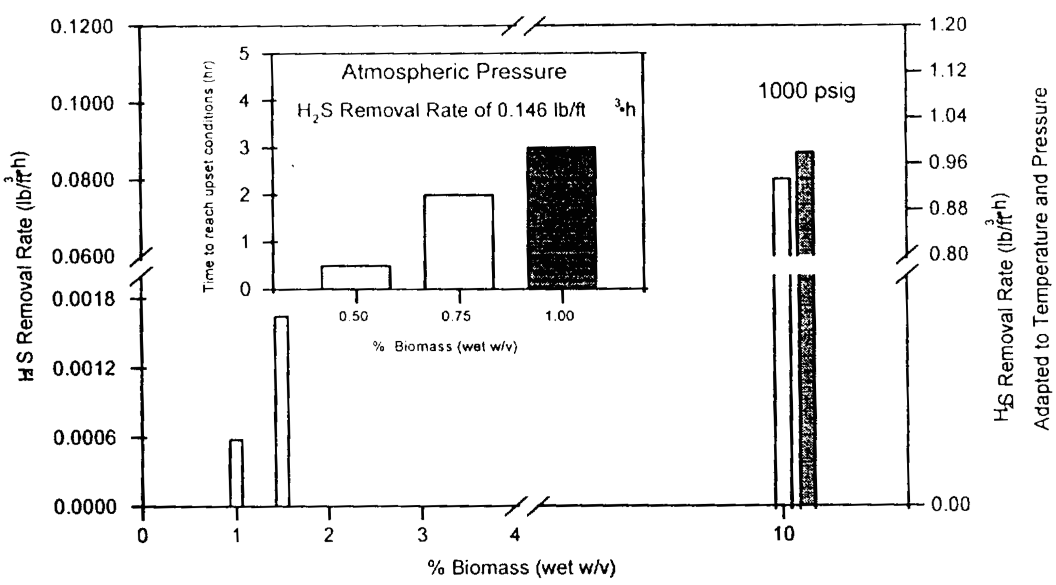


Removal of H_2S from a Gas Composition of 10,000 ppm H_2S, 10% CO_2 in N_2, in a Parr Pressure Reactor in Batch Mode at 1000 psi Containing Different Sources of Organic Nitrogen with 10% (Wet w/v) SS II

Biomass Concentration Effects The biomass loading rates is very important from both, the process efficiency, as well as the cost-effectiveness, points of views. While, higher biomass loadings can result in higher reaction rates, it can also pose processing problems in terms of pumping, cost of pumps as well as the higher energy requirements. All these factors would ultimately affect the process cost. Preliminary data from the microbioreactors and the data at the atmospheric pressure indicates that with an increase in the biomass loading from 0.5% to 1.0% (wet weight of cells to volume of the liquid in the reactor), the rate of biodesulfurization of H_sS from the SSNG increases from about 0.05 to 0.146 lb•ft^3hr^{-1}. With this background, the effect of biomass loading at high pressure was also evaluated. Since the results from experiments at 1 psi, already established a base line of 1% (wt/v) microbial loading, the effect of 10% biomass loading at 1,000 psi was evaluated. The data shows that at a pressure of 1,000 psi and of the temperature of 140°F (60°C), the SSII microbial consortium removes the H_2S at a rate of 1.0 lb•ft^3hr^{-1} (Figure 3), which is 10 fold higher than that obtained by the Scavenger Technologies. This, the data presented in Figure 3, establishes:

1. a direct relationship between biomass loading and H_2S removal rates,
2. the removal is independent of the pressure of one atmosphere or 1,000 psi.

Figure 3: Higher Biomass Loadings (up to 10% by wet weight of bacterial Cells) linearly Enhance H_2S Removal Rates Independent of the Pressure

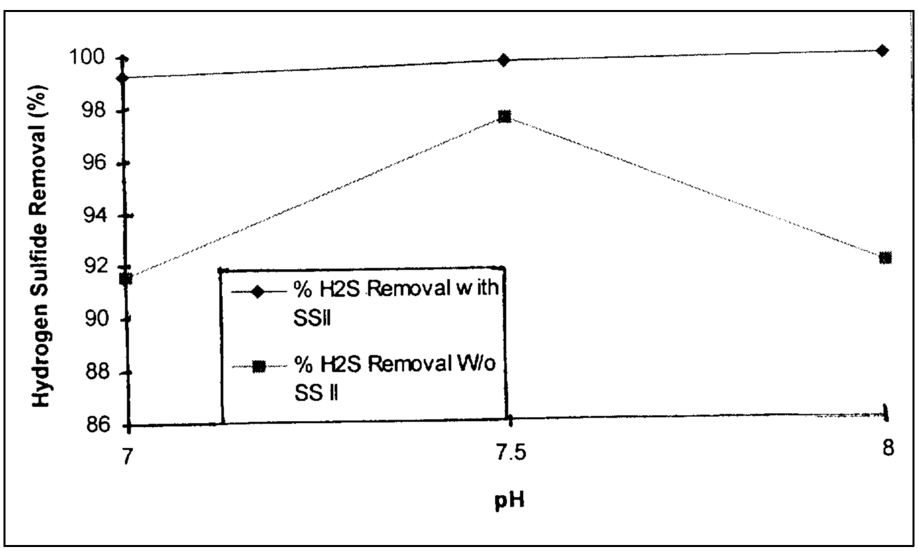


Increase in Biomass Loading of up to 10% Enhances the H_2S Removal Rates Independent of the Pressure

Effect of pH The other parameter having a strong bearing on the process performance and the cost efficiency is the process pH. Preliminary data showed that the highest removal of the sulfur species are obtained when the pH of the liquid in the Parr reactor is initially at 7.5 to 8 (Figure 4). Later experiments at both, atmospheric pressure and 1,000 psi have confirmed these data. Data from experiments run in semi-continuous mode and up to 100 hours, however, show that during the removal process the pH of the liquid in the Parr reactor drops and at the end of the 100 hr, the pH was found to be between 6.5 and 6.6. This information is consistent because an acidic gas is constantly being pumped into the system.

***Effect of Temperature*:** Temperature is an important parameter when dealing with micro-organisms that have essentially been derived from ambient environments. The BIODESULF™ process has been tested for performance in the temperature range of 50°F (10°C) to 140°F (60°C). While the data presented in Figure 5 clearly indicate a linear relationship between the temperature and the H_2S removal rates by the microbial consortium used in the BIODESULF™ Process, the actual range needs to be further investigated. This necessity is because of the different conditions of temperature under which the process might have to work. These conditions might range from < 50° F in nights to > 152° F in day time in deserts. Despite this requirement, the data obtained thus far, establish that the rate of H_2S removal by the microbial consortium serving as biocatalyst in the BIODESULF ™ Process is 1.0 lb/ft^3hr^{-1} (Figure 5). This rate is 10 fold higher than that obtained by the Scavenger Technologies[4,9,15-17]. Based on the removal rate obtained in the BIODESULF™ Process, M.W. Kellogg personnel have independently conducted an economic study of the BIODESULF™ Process. According to their report[15], for treating 1 TPD sulfur, the BIODESULF™ requires 600 gallons of liquid reagent in contrast to 4000 gallons for the ARI- LO-CAT™ and 10,000 gallons for the BIO-SR™. A cursory analysis

Figure 4: Preliminary Studies Showed Highest H_2S Removal at pH of 7.5 to 8

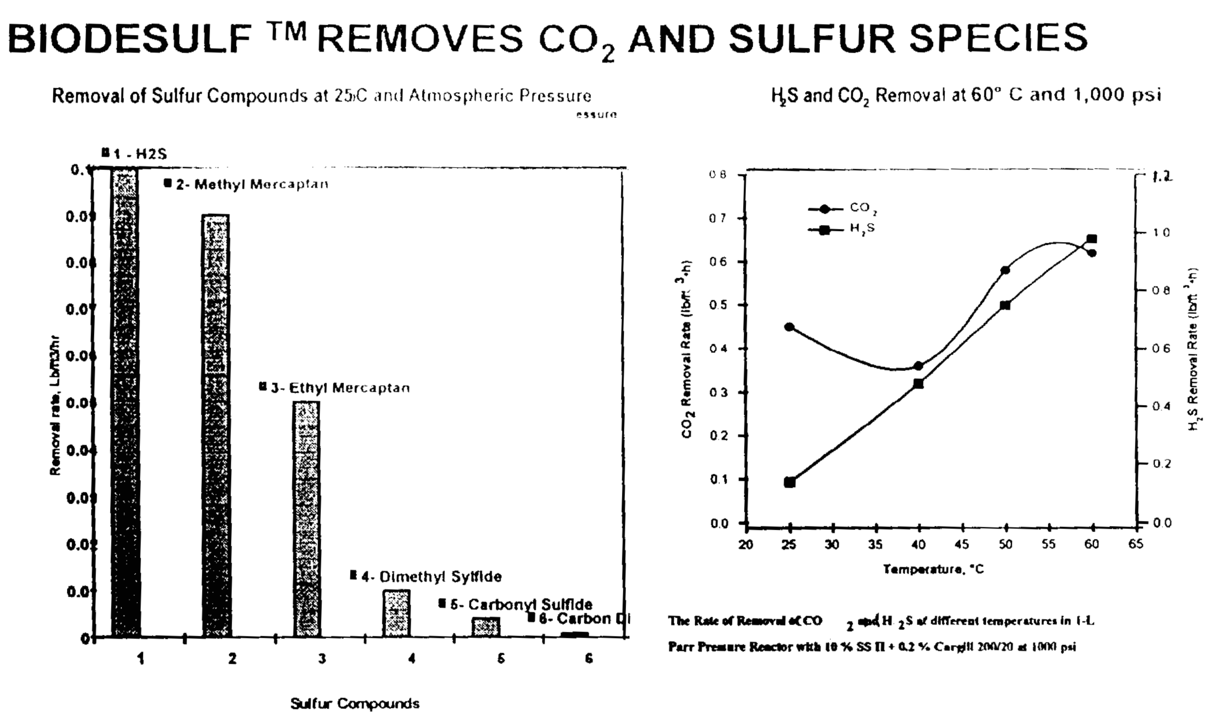


of the conclusions from the M.W. Kellogg report means that the BIODESULF™ would potentially be:

- 6.7 fold more cost-effective than the ARI-LO-CAT™, and
- 16.7 fold cost effective than the BIO-SR™.

Range Of CO_2 Concentration One of the recommendations of the M.W. Kellogg report[15], was also to evaluate the range in which the CO_2 present in SSNG is removed by the SSII microbial consortium. Therefore, the experiments conducted to evaluate the removal rates of H_2S were repeated. This time, however, in addition to evaluating only the H_2S removal, at the same time points as those taken for the H_2S, additional headspace samples from the Parr reactor were analyzed on a GOW-MAC Gas chromatograph equipped with TCD. The data (Figure 5) clearly indicate that at higher temperatures (up to 140°F, 60°C), not only is the sulfur removal enhanced, but also the CO_2 removal is increased. These data have an important bearing on the overall process economics. Thus, in one step, one can remove a number of contaminants (H_2S, CS_2, dimethyl, ethyl and methyl mercaptans and CO_2) from the SSNG and thus enhance the calorific value of the processed SNG, which is now sweetened. These data further support the hypothesis that under anaerobic conditions, the chemoautotrophic bacteria utilize CO_2 as the source of carbon[2,5,19]. Thus, the possible mechanism of bioconversion of reduced sulfur species (eg. H_2S) to elemental sulfur by the SSII microbial consortium might involve the utilization of H_2S as a source of energy and CO_2 as a source of carbon, when either NH_4^{+1}, or NO_3^{-1}, or both are present in the culture medium as is the case for the BIODESULF™ Process.

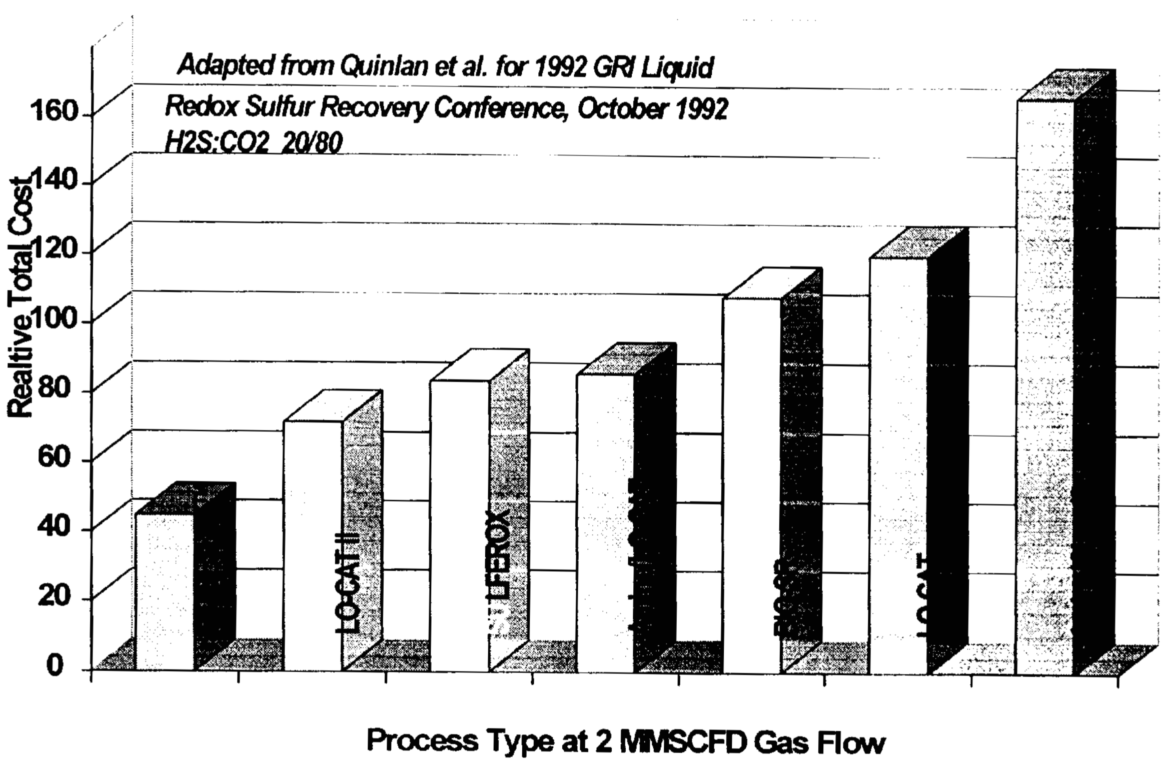
Figure 5. At a Higher Temperature, Both CO_2 and H_2S Revomal is Enhanced



Thiosulfate Requirements Another important parameter for the BIODESULF™ Process is also related to the metabolism of the microbial consortium as well as to the basic biochemical mechanism. The microbes require thiosulfate ion to initiate their activity. Thiosulfate can be an expensive ingredient of the chemicals cost for the overall process. A series of experiments were run to investigate the amount of thiosulfate required to prepare the microbial inoculum for the removal experiments. Thiosulfate concentrations of 1% to 3% (W/v) were examined. Data (Figure 6) indicate that initial thiosulfate concentrations >1.5% are inhibitory to microbial growth and can be detrimental to the process performance. On the basis of these results, the cost effectiveness of the process (Figures 6 and 7) can be further enhanced.

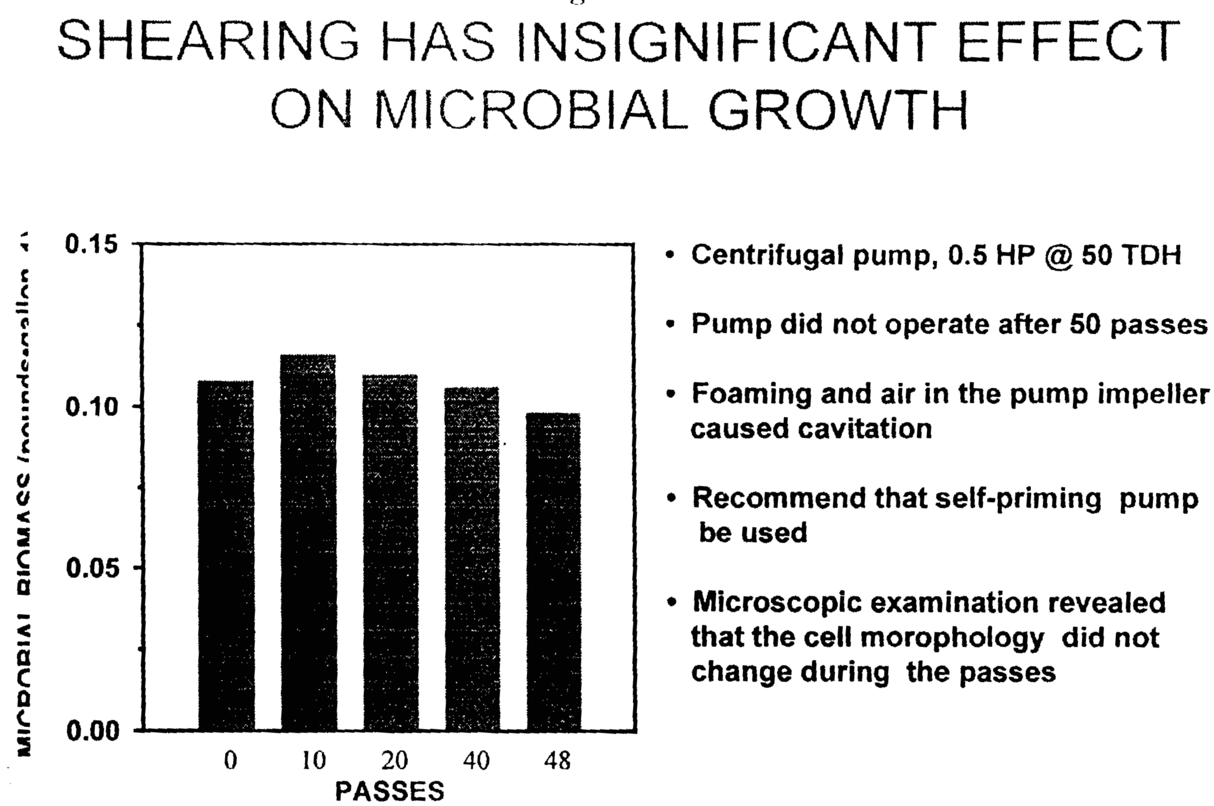
Process Economics M.W. Kellogg personnel have been performing the economic analyses of a number of processes for GRI[14,16]. The assumptions that they used for making these calculations have recently been reviewed and used by Rehmat et al.[16] to calculate the economics for the BIO-SR™ Process. Using the same assumptions, and the data obtained from different experiments in the study reported here, the preliminary economics for the BIODESULF™ process was calculated based, also on the analysis of the BIODESULF™ process by M.W. Kellogg[15]. On this basis, the BIODESULF™ process is least costly (Figure 7) of all the processes being used or developed for processing the SNG at least up to the production level of 2 MMSCFD.

Figure 7: The BIODESULF™ Process is Cost-Effective



Effect of Shear on Microorganisms The effect of shear stress on microorganisms during pumping is of concern. This was evaluated in the laboratory by pumping the cell suspension of SSII consortium through a centrifugal pump. A volume of 11 of the bacterial suspension at 10% (wet weight of cells/volume of liquid) microbial loading was pumped at rate of 0.33 l min^{-1}. On this basis 10 passes were completed in 3.33 mins or 3 mins and 20 seconds. Aliquots of cell suspension containing the biomass were collected after 10, 20, 30 40 and 48 passages through the pump. These aliquots were then reinoculated into fresh nutrient medium for growing the micro-organisms. The data on growth (Figure 8) indicate that shear force has insignificant effect on microbial growth and survival. This data is in agreement with the literature reports[21] that active microbial populations survive at different shear forces as evidenced by presence of active micro-organisms isolated from the deep sea vents. The removal rates from shear treated microorganisms is yet to be investigated.

Figure 8:



Future Work

The data obtained thus far, clearly indicate the potential of the BIODESULF™ Process for the removal and mitigation of sulfur species and CO_2 from Low Quality/Sour Natural Gas. The planned future work includes:

- maximizing the mass transfer rates through the evaluation of different designs of sparger systems including:
    - ♦ branched tee,
    - ♦ spiral and fritted disc, and
    - ♦ a straight tube bubbler of the type used in the prior work as a baseline.

- establishing the minimum and maximum range of operations for:
    - ♦ biomass loading vs. thiosulfate requirements,
    - ♦ temperature and pH, and
    - ♦ maximizing the removal rates for mercaptans.
- modifying the conceptual flow scheme for a skid-mounted 50-l pre-pilot demonstration reactor,
- modifying design of a 50-l pre-pilot reactor and collecting performance data on this reactor.

Applications/Benefits

Conventionally, Amine/Claus Process have been used for the desulfurization of Sour Natural Gas. While highly efficient in removing the H_2S at large throughput volumes, the major limitation of these processes is dramatic cost-prohibitiveness when treating low volume (1-5 MMSCFD). Moreover, in these processes, the sulfur species are merely removed from the gas stream, requiring a back-end system to further treat the concentrated sulfur species in another compatible system. Similarly, LO-CAT systems are cost effective in the gas input range of 5 to 30 MMSCFD. In contrast to these prior technologies, the BIODESULF™ provides following benefits:

1. one step removal and mitigation of H2S , COS, mercaptans and potentially other sulfur species commonly encountered in the Sour Natural Gas stream,
2. reduce the CO_2 content of the Sour Natural Gas,
3. microbial catalysts are self-regenerating, requiring less operating cost,
4. less labor intensive process,
5. temperature, pH and pressure conditions for the operation of the process are compatible to those commonly encountered in the gas processing industry, therefore, retrofit is potentially possible, and
6. process can be easily and cost-effectively scaled down in the range of $\leq$ 1 MMSCFD to 2 MMSCFD, with potential for up to 5 MMSCFD.

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