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**EVALUATION OF AVAILABLE
MHD SEED-REGENERATION PROCESSES
ON THE BASIS OF ENERGY CONSIDERATIONS**

by

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U of C-ADA-USDOE

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Chemical Engineering Division

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ABSTRACT

Of the several processes described in the literature that are capable of separating sulfur from alkali-metal sulfates, seven processes were selected as candidates for regenerating seed material for reuse in open-cycle MHD. After a brief assessment of each process, two were selected for a detailed analysis, namely, a process developed by the Pittsburgh Energy Research Center (PERC) and a modified version of the Tampella process. The processes were compared on the bases of energy requirements and the amount of research work needed to develop a seed-regeneration process for MHD systems. The energy requirements given should be considered as rough values, because factors such as heat losses and component efficiency were not included in the analysis. On the basis of energy consumption, the PERC process has a slight advantage over the Tampella process; on the basis of the present state of development of various components, the Tampella process has a clear advantage. Accordingly, it was recommended that developmental programs be carried out for both the PERC and Tampella processes.

I. INTRODUCTION

The open-cycle, coal-fired MHD power system, in addition to having a higher efficiency than other fossil-fueled power systems, has the advantage of a self-contained sulfur-removal capacity. The potassium seed plays a dual role, first, by increasing the electrical conductivity of the hot combustion gases by thermal ionization and, second, by eliminating SO_2 from the gaseous effluent. The spent seed is collected in various downstream components, predominantly as a mixture of water-soluble salts, K_2CO_3 and K_2SO_4 , contaminated with fly ash. Because of the high cost of seed (about \$0.20/lb for K_2CO_3 and about \$0.05/lb of K_2SO_4), and the large quantities of seed required, a potassium throw-away system for sulfur removal is possible only if there is an adequate supply of K_2CO_3 and sufficient demand for K_2SO_4 . At present, it is not economically feasible to consider a potassium throw-away system; therefore, to exploit the desulfurization capability of the MHD seed material, sufficient quantities of sulfur must be separated from the recovered K_2SO_4 to allow the seed to be reused.

Several processes described in the literature are capable of separating sulfur from alkali metal sulfates but at present none are directly applicable to MHD seed recovery. Some processes have never been fully developed and most of the commercial processes either were developed especially for sodium salts or are not easily adaptable to an MHD system. Data for engineering design and development of a complete seed-regeneration system for MHD are either not available, or are incomplete.

This report describes a study performed at Argonne National Laboratory (ANL) to evaluate available processes and recommend the most promising for further development. Concurrently, a similar but independent evaluation was conducted at the University of Tennessee Space Institute.

To maintain uniformity, a common set of conditions and assumptions was adopted for both evaluations. These are summarized in Table 1.¹

Table 2 lists the characteristics of Illinois #6 coal, which provided a uniform coal composition for both evaluations.

Other constraints imposed on the evaluations were as follows: (1) flue-gas scrubbing processes and processes yielding sulfur in the form of a waste solid compound (e.g., $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) were to be excluded from consideration and (2) the sulfur recovery step was to be carried out only to the stage of elemental sulfur.

Calculations showed that, for the conditions adopted for the evaluation, about 85% of the K_2SO_4 would have to be converted to K_2CO_3 to restore the desulfurization capability of the MHD seed.

Table 1. Conditions and Assumptions Used in Evaluation.

Size of plant:	1000 MWt
Net electrical efficiency:	50%
Type of coal:	washed Illinois #6
Sulfur content of coal:	3 to 3.5 wt %
Seeding level:	1 wt % in flue gases
K/S ratio:	at least 10% excess over that required to react with sufficient sulfur in the coal to meet EPA emission limits (1.2 lb SO ₂ /10 ⁶ Btu).
Overall efficiency for potassium recovery:	95%
Composition of spent seed mixture:	95 wt % K ₂ SO ₄ + K ₂ CO ₃ , 5 wt % fly ash

Table 2. Characteristics of Washed Illinois #6 Coal
(Source: Burning Stone #1 Strip Mine)^a

Proximate Analysis, wt %		Ultimate Analysis, wt %	
Moisture	12.05	Moisture	12.05
Ash	10.40	Ash	10.40
Volatile Matter	36.05	Sulfur	3.40
Carbon	41.50	Carbon	60.72
		Hydrogen	4.22
		Nitrogen	1.14
		Chlorine	0.02
		Oxygen	8.05
Ash Analysis (ignited basis), wt %		Heating Value: 11,100 Btu/lb Free Swelling Index: 3.0 Hardgrove Grindability: 56	
P ₂ O ₅	0.08		
SiO ₂	50.83		
Fe ₂ O ₃	21.13		
Al ₂ O ₃	18.33		
TiO ₂	0.81		
CaO	3.20		
MgO	0.96		
SO ₃	2.30		
K ₂ O	1.93		
Na ₂ O	0.38		
Undetermined	0.05		
Total	100.00		

^aSee Ref. 2.

In the evaluation study reported here, seven processes for separating sulfur from alkali metals were selected for assessment: (1) the PERC process (developed by the Pittsburgh Energy Research Center),³ (2) the Aqueous Carbonate (AC) process,⁴ (3) the Tampella process,⁵ (4) the Markant process,⁶ (5) the Engel-Precht process,⁷ (6) the Formate process,⁸⁻¹⁰ and (7) the Double-Alkali process.⁹ On the basis of the above-mentioned ground rules, some of the processes were ruled out. Detailed mass and heat balance calculations were carried out for the PERC and modified Tampella processes. The calculated results were then utilized to compare these two processes with respect to their energy requirements. Two processes were considered for sulfur recovery: the Claus process¹¹ and the Stretford process.^{11a} The latter process was selected for sulfur recovery following the seed regeneration. Owing to inadequate information on the Stretford process, the sulfur recovery step was included in the mass balance calculations but not in the heat balance calculations.

Because kinetic and thermodynamic data for the selected processes are unreliable and/or incomplete, economic evaluations and comparisons are difficult and inconclusive. For example, the University of Tennessee Space Institute (UTSI)^{11b} estimated that the cost of regeneration by the PERC process will be about 5.68 mills/kWh for a 1000 MWe MHD power plant using coal containing 4.1 wt % sulfur. In a similar study of the PERC process, Westinghouse¹² estimated a cost of about 1.70 mills/kWh for a 220 MWe MHD power plant using 0.8 wt % sulfur coal, with a cost breakdown of 25% for fixed cost of gasifier and reactors, 32% for fixed cost of equipment other than the gasifier and reactors, and 43% for operating costs and cost of coal. When we adjusted the Westinghouse estimates for a 1000 MWe plant and 4.1 wt % sulfur coal, the seed regeneration cost was 5.17 mills/kWh; the cost breakdown in this case, was 12% for fixed cost of gasifier and reactors, 15% for fixed cost of equipment other than gasifier and reactors, and 73% for operating costs, mainly for coal. Although the 5.17 mills/kWh figure agrees well with the UTSI estimate of 5.68 mills/kWh, it can be seen from the cost breakdowns that seed regeneration cost for small plants is sensitive to the cost of the reactors and gasifier, and for larger plants it is sensitive to the gas utilization. The Westinghouse cost and our adjusted value of 5.17 mills/kWh were based on 21% gas utilization; however, PERC¹² has used gas utilizations as high as 85% in their studies. When the cost value based on the Westinghouse estimate was further adjusted for 85% gas utilization, thus reducing the capital cost of the gasifier and the cost of coal, the seed regeneration cost was only 2.09 mills/kWh. Thus, knowledge of reaction rates, gas utilization, and chemical equilibria is very important in making reasonable cost estimates. Information on materials of construction, component design, and fabrication are also important. However, these kinds of information were not available for the seed regeneration processes selected for this study; therefore, economic comparisons seemed unjustified. Instead, our evaluations were based primarily on the technical feasibility of a process, its stage of development, and compatibility of the process with the environment.

The MHD development program sponsored by DOE has the goal of commercial MHD demonstration by the late 1980's. The first major milestone in meeting this goal is a pilot-scale Engineering Test Facility (ETF). As a part of the complete plant, ETF should contain seed-recovery and -regeneration systems. Westinghouse Electric Corp. and AVCO Everett Research Laboratory, Inc. have proposed conceptual seed recycle systems for ETF. In their work, both the

contractors have critically reviewed some of the available seed regeneration processes. Information given in these two contractors' preliminary reports^{9,12} has been considered for the evaluations in this report.

In this report, most of the units used in calculations are those commonly used in industrial and engineering technologies. For the convenience of readers who may wish to perform calculations in metric (SI) units, appropriate conversion factors are given in the Appendix.

II. AVAILABLE SEED-REGENERATION PROCESSES

From among the various applicable processes described in the literature, the following seven processes were selected as possible candidate processes for seed regeneration.

A. PERC Process

The chemical reactions involved in the PERC³ process are as follows:

From the limited data obtained at the Pittsburgh Energy Research Center (PERC), values for the percentage gas utilization were calculated and reported by AVCO Everett Research Laboratory.⁹ AVCO calculated that for reductants of pure hydrogen and pure carbon monoxide, both at 750°C, the gas utilization values per pass were only 2.5 and 2%, respectively. For the regeneration step at 500°C, AVCO calculated that for a mixture of 50% CO₂ and 50% steam only 3.9% of the CO₂ was utilized per pass. However, with mixtures of CO and H₂, coal, producer gas, etc. and different reactor designs with gas recycle and stage-wise operations, it may be possible to improve the gas utilization in this process. Theoretical equilibrium calculations indicate the possibility of obtaining higher gas utilizations (up to about 90%). A coal gasifier is required in this process; however, extensive cleaning of the gasifier effluent to remove CO₂, tarry materials, and particulates is not required. The main disadvantage of this process is that it requires three high-temperature operations, namely, coal gasification, seed reduction, and seed carbonation/oxidation. Another possible problem is caused by phase changes during the reduction step, which could cause the reaction mixture to melt and then resolidify.

B. Aqueous Carbonate Process

The Aqueous Carbonate (AC) process was developed and has been tested in a pilot plant for a sodium system by Atomics International.⁴ A modified version for the potassium system would have similar chemical steps, as indicated below.

In reducer In precarbonator

The entire aqueous solution containing potassium salts is transferred to the carbonator, where the following reactions occur:

Liquid effluent from the carbonator, upon evaporation/crystallization, yields K_2CO_3 .

A recent Westinghouse report¹² points out that the estimated capital cost for a plant handling 10 kg/s of K_2SO_4 , and using the AC process to completely convert sulfate to carbonate will be $\$44.1 \times 10^6$. For a plant handling 1.25 kg/s of K_2SO_4 (about 192 Mwt size) and using Illinois coal in the MHD combustor, the net energy requirement is estimated⁹ to be 26.6×10^6 Btu/hr. Thus, for the AC process the required energy consumption and capital cost are both large.

Water quenching of the melt, as is prescribed for this method, requires the use of large amounts of water to prevent vapor explosions. The use of coal in the reducer has not yet been demonstrated. If coal or coke containing ash were used in the reducing step instead of low-ash petroleum coke, an additional 1-3% of the potassium would be lost as unextractable silicates. The use of molten salt in the reducer results in serious materials problems.

C. Modified Tampella Process

The Tampella process,⁵ as developed and commercially tested for the sodium system in the paper and pulp industry, involves only the carbonation of sulfide. To employ a modified version for a seed-regeneration system, an appropriate step to reduce K_2SO_4 to K_2S is thus required. The chemical reactions involved in the modified version for the potassium system are the following:

In reducer In precarbonator In stripper

In carbonator

With the addition of a reduction step, the Tampella process is quite similar to the AC process. The main difference is that the AC process uses the same unit for the stripping of H_2S and formation of potassium carbonate and bicarbonate, whereas the Tampella process has a separate section for stripping of H_2S and formation of potassium bicarbonate. Consequently, the H_2S concentration in the effluents from the AC process is much lower than in the Tampella process.

For the reduction step, the paper and pulp industry employs a kraft or similar type of furnace, into which spent sodium sulfite liquor (also called black liquor) is blown through a series of spray nozzles. To burn lignins contained in this liquor, air is introduced at various levels inside the furnace. Sodium salts coming out of the nozzles solidify on the opposite walls where the reduction of sodium sulfite and sodium sulfate to sodium sulfide takes place. Carbon from the lignins serves as the reducing agent. The reduced mass slumps slowly to the bottom of the surface, where it is collected in the molten condition and then removed. Thus, in the paper and pulp industry, the reducing agent, the heat source, and the material to be reduced are all present in one stream. In the MHD system, the material to be reduced and the reducing agent are not necessarily present in the same stream. Most likely, coal or coke will be preferred to reduce K_2SO_4 to K_2S ; therefore, a different furnace design from that of the kraft furnace may be required. In a blast furnace concept, coal/coke along with solid or molten K_2SO_4 would be fed to the top of a shaft furnace; an air blast would be introduced into the bottom of the shaft through suitable tuyeres, and molten slag and K_2S would be withdrawn from the bottom. In a spray burner concept, as in the kraft furnace, finely ground coal/coke would be mixed with molten K_2SO_4 (or with a saturated aqueous solution of K_2SO_4) and blown into the furnace through a series of spray nozzles. Alternatively, powdered carbon and K_2SO_4 could be blown through separate nozzles, with the two streams impinging on each other. A third concept would be to use a two-compartment, molten-salt reactor, as was used in an earlier version of the AC process.⁹

No kinetic or experimental data are available for the potassium system. If coal or coke was used in the reduction furnace to reduce K_2SO_4 in addition to normal system losses, some additional potassium would be lost because of the reaction with ash constituents, such as SiO_2 , Al_2O_3 , etc. As in the AC process, the materials problems with the potassium system could be very significant for the reduction furnace. Problems of vapor explosions in water-quenching of the melt also exist in the Modified Tampella process.

D. Markant Process

The chemical reactions involved in the Markant process⁶ are as follows:

This process has not been completely tested or developed. Sufficient conversion of K_2S to KOH , and complete separation of zinc and potassium may be problems. The sulfides of potassium and zinc are known¹³ to form a complex, $\text{K}_2\text{S} \cdot 3\text{ZnS}$, which on exposure to air forms potassium thiosulfate. Thus, in the second step of the process, some K_2S may be lost by this mechanism. Also, it has been reported¹⁴ that ZnO , after regeneration form ZnS , does not regain its original chemical reactivity. If coal or coke is used instead of oil as a heat source and reducing agent, the ash constituents such as SiO_2 , Al_2O_3 , CaO , etc. may react with K_2SO_4 causing an additional seed loss. The chemical reaction for reduction of K_2SO_4 to K_2S is the same as that of the AC and Tampella processes, although the conditions for the reduction are considerably different. However, the Tampella or AC process does not require an additive such as ZnO for the sulfur-separation step. The Markant process seems to have no clear advantage over the Tampella or the AC process, except perhaps a higher concentration of sulfur in the gaseous effluent. In the Markant process, most of the sulfur is released in the form of SO_2 while in the AC and Tampella process it is in the form of H_2S .

E. Engel-Precht Process

The chemical reactions in the Engel-Precht process⁷ are as follows:

This process has been used industrially in Germany for many years to produce K_2CO_3 from KCl or K_2SO_4 . The main advantage is that the process is not energy intensive; it consumes fuel only to produce MgO (probably from dolomite) and needs very small amounts of heat for the second step. However, the theoretical overall conversion of K_2SO_4 to K_2CO_3 is between 50 and 70% (which is insufficient for high-sulfur coal), and the loss of potassium in liquid waste stream is about 5%. Unless MgSO_4 can be utilized for its chemical value, MgSO_4 presents a waste-disposal problem because it is water-soluble, and thus can be leached by groundwater.

F. Formate Processes

The chemical reactions for the Formate process⁸⁻¹⁰ are as follows:

This process was used industrially in Germany until at least the beginning of World War II. Some K_2SO_4 may be lost as $CaSO_4 \cdot K_2SO_4 \cdot H_2O$ (syngenite) or as $5CaSO_4 \cdot K_2SO_4 \cdot H_2O$. According to a recent AVCO report,⁹ sulfur recovered in the form of gypsum ($CaSO_4 \cdot 2H_2O$) in this process is gel-like and difficult to filter. On the basis of work done on the sodium system,¹⁵ the AVCO report recommends solving the filtration problem by recycling sufficient formate solution so that the potassium sulfate solution entering the reactor will contain at least 10% formate. Most of the information about the formate process is patented and not available in open literature. However, from the available information, it is evident that the process can provide K_2CO_3 of 99% purity with overall conversion of K_2SO_4 to K_2CO_3 greater than 90%.¹⁰

The AVCO report⁹ also points out that the second step of the process could be avoided by feeding potassium formate, as a dry powder or a concentrated solution, to the MHD combustor, with no significant penalty on plasma conductivity. By feeding the seed material to the MHD combustor as potassium formate, the oxidation step to carbonate would take place inside the combustor, and the heat of this exothermic reaction (109.9 kcal/mol at 298 K) would be recovered by hot combustion gases. This recovery might reduce the overall process energy requirement.

Carbon monoxide required for the Formate process is usually obtained from a coal or coke gasifier. According to the AVCO report,⁹ tarry materials and especially CO_2 must be removed from the gasifier effluent before it enters the reactor. Otherwise, the CO_2 reacts with lime to form $CaCO_3$, which then causes a scaling problem in the evaporators; also, tarry materials condense on the walls and affect the heat and mass transfer.

G. Double Alkali/Causticization/Hydrated Lime Processes

The chemical steps involved in these similar processes⁹ are as follows:

Owing to the limited solubilities of calcium salts and the thermodynamic equilibrium of the system, very dilute solutions of K_2SO_4 (about 1.4×10^{-2} mol/L) are required for greater than 75% conversion to KOH in the normal operating temperature range of 150 to 190°C. Using Illinois #6 coal this means that subsequently about 400 lb of water used for dilution needs to be evaporated per pound of K_2SO_4 that is processed. Some K_2SO_4 may be lost as a double salt with $CaSO_4$. Sulfur recovered in the form of $CaSO_4 \cdot 2H_2O$ poses a waste-disposal problem. Calcium remaining in solution with the potassium can cause scaling problems in the evaporator due to formation of $CaCO_3$.

III. PROCESS CONSIDERATIONS

Each of the seven processes described above was assessed using the conditions and assumptions described previously in Section I.

The Engel-Precht process, although having an advantage of being a low-temperature process was eliminated from consideration for several reasons. First, according to the ground rules, sulfur from K_2SO_4 was to be recovered in the form of elemental sulfur and no sulfur throw-away processes were to be considered. Other reasons were as follows: (1) theoretical conversion of K_2SO_4 to K_2CO_3 is only 50-70%, which is inadequate for Illinois coal; (2) about 5% K_2SO_4 could be lost with the $MgSO_4$ solution in the waste stream, and (3) $MgSO_4$ presents a waste-disposal problem because it is water-soluble and therefore can be leached by groundwater.

The Formate process was not considered further because it also did not meet the ground rules of sulfur recovery. This process also has the disadvantage that it requires pure CO to prevent scaling in the evaporators. Consequently, extensive purification is required to remove CO_2 and tarry materials from the gasifier effluent.

The Double Alkali process also did not meet the requirement of sulfur recovery. An additional disadvantage is the necessity for using very dilute solutions of K_2SO_4 to achieve the necessary conversion to hydroxide. As mentioned earlier, to achieve 75% (or greater) conversion to KOH would require the evaporation of about 400 pounds of water for each pound of K_2SO_4 used.

The Markant process qualified for a more extensive analysis. The first step in the Markant process, namely, the reduction of K_2SO_4 to K_2S , is very similar to the reduction steps in the modified Tampella and the AC processes. However, with the present oil shortage, it may not be economical nor possible to feed K_2SO_4 to the furnace in a form of an oil slurry, as the Markant process requires. Once the reduction melt of K_2S is formed, the subsequent carbonation to K_2CO_3 can easily be carried out following the established techniques of the Tampella process. The use of ZnO to scavenge sulfur out of K_2S may subsequently give concentrated SO_2 by roasting of ZnS; however, this procedure would have to be developed and demonstrated. Very little is known about the double salt, $K_2S \cdot 3ZnS$; therefore, a detailed study would be required to establish conditions under which double salt formation can be avoided or minimized. On air oxidation, this complex forms thiosulfates and thus reduces the overall conversion from sulfate to carbonate. Thus, it is doubtful that the Markant process can achieve adequate conversion of K_2S and sufficient recovery of ZnO. Another problem is that upon regeneration ZnO does not completely regain its original chemical reactivity.

The differences between the modified Tampella process and the AC process are minor. In the AC process, because the stripping and carbonation steps are carried out in a single unit, the concentration of H_2S in the effluents is lower than in the Tampella process. The AC process has been tested so far only on a pilot-plant scale for a sodium system. Although most of the components in the AC process have been tested separately, the integrated process, as an entire system, has not yet been tested. Also, the use of coal to reduce Na_2SO_4 to Na_2S has not yet been demonstrated. Because very little information on the AC process is available in the literature, the operating and design conditions for the various components are not known. On the other hand, the Tampella process has been demonstrated to be a practical system for sodium compounds by the paper and pulp industry. Available information on the operating and design conditions for the Tampella process, although limited, was considered to be helpful in designing a system for potassium; therefore,

a decision was made to carry out a detailed analysis based on the modified Tampella process. Information on the extensive operation of kraft-type recovery furnaces should also provide valuable input for the design of a reduction furnace for the seed regeneration system.

Further information on the Aqueous Carbonate process may become available in the future, as the result of a planned project of the Environmental Protection Agency (EPA) and Empire State Electric Energy Research Corporation (ESEERCO).^{15a} These organizations are jointly sponsoring a 100-MW demonstration plant to be operated at the Huntley generating station of Niagara Mohawk Power Corporation (NMPC) on one of their coal-fired boilers; the AC process will be tested as part of the flue-gas desulfurization process to determine its capability for regenerating the SO_2 -absorbing media. If the results of this demonstration are favorable it may be possible in the future to direct the developmental program on seed regeneration toward this process with little difficulty.

Of the various seed regenerating processes, the Westinghouse report¹² recommended the PERC process on the basis of three features: (1) minimum recycle of water with the regenerated seed, (2) minimum energy requirements, and (3) minimum capital requirements. In their opinion, the PERC process is relatively easy to control. In combination with the Claus or Stretford process, the PERC process also provides the sulfur in the desirable form of elemental sulfur. However, the PERC process has many disadvantages, such as apparently poor gas utilization, three high-temperature operations (coal gasification, seed reduction and seed carbonation), and unknown kinetics. From equilibrium calculations, better gas utilization seems possible and, with proper reactor design, gas recycle, and stage-wise operation, gas utilization may be improved. Since experimental information about this process is incomplete, it should remain under consideration as a possible seed regeneration process until more engineering data are obtained.

The evaluations of the Tampella and PERC processes were conducted on the basis of process feasibility and energy usage only. Cost analyses were not included because data for making reasonable estimates were not available. Although the Tampella process is a proven commercial process it has been used only for sodium salts. The PERC process has not been developed beyond the laboratory stage, and no reliable kinetic data are available. Without these data the principal items or equipment for a seed-regeneration system (e.g., the reducer and regenerator) cannot be designed nor can their cost be estimated. Moreover, corrosion and erosion by high-temperature potassium salts may also affect construction costs. We have included pertinent results of other published economic analyses, however, and a brief discussion of these is presented.

IV. SULFUR-RECOVERY PROCESSES

Effluents from seed-regeneration systems based on the PERC, Tampella, and AC processes will contain H_2S and small amounts of COS , CS_2 , SO_2 , S_2 , etc. Effluents from the Markant process will contain mainly SO_2 . Environmental and safety standards require that most of these compounds be removed from waste gas streams prior to discharge. Various sulfur recovery processes are described in the literature; some are designed to operate on the tail gases

(1-3%, H₂S) and some on the concentrated feed (5-20% H₂S). The Claus, Trentham Trencor-M, and Stretford processes are described as being capable of handling concentrated feed. The Beavon, Scot, Wellman-Lord, IFP, Cold Bed Absorption (CBA), and Sulfreen processes are described as operating mainly on the low-sulfur tail gases from the Claus plant.

In reference to seed regeneration systems, interest will be mainly on processes capable of handling concentrated H₂S feeds. Because the Trentham Trencor-M process has not yet been demonstrated successfully on a commercial unit, only the Claus and Stretford processes were considered.

Of the processes capable of recovering sulfur from SO₂, the Resox process¹⁶ has been well developed. In this process, in the presence of steam, SO₂ is reduced by coal (preferably anthracite coal) at about 1100-1500°F yielding CO₂ and sulfur. This process can handle SO₂ concentrations from 2 to 20 vol %. It can tolerate residual fly-ash and eliminates gas drying. The absence of catalysts in this process sidesteps the concern over trace contaminants. Furthermore, the Resox process offers an interesting flexibility of recovering sulfur as H₂S rather than an elemental sulfur-rich stream. Because of the incomplete development of the Markant process, a sulfur recovery step appropriate to other steps in the process has not yet been tested. However, in the future, if more work is to be conducted on the Markant process, then the Resox process is certainly a strong candidate for sulfur recovery.

A. Claus Process

The Claus sulfur-recovery process¹¹ is a multiple-stage, catalytic process used for converting H₂S to elemental sulfur by following the Claus reaction:

The sulfur dioxide required for the reaction is produced by burning about one-third of the H₂S present in a furnace ($\text{H}_2\text{S} + 3/2 \text{O}_2 \rightarrow \text{SO}_2 + \text{H}_2\text{O}$). The feed to a conventional Claus plant should contain at least 20% H₂S; below this level it becomes impossible to maintain a stable flame temperature in the furnace. For acidic gases containing about 15 to 20% H₂S, additional heat can be added to the system by preheating furnace combustion air to about 700°F (370°C). At H₂S concentrations below 15%, some of the product sulfur can be recycled and burned in the furnace; this step extends the range of H₂S concentrations that can be handled in Claus feed plants to feeds down to about 5%.

The most severe limitation on Claus performance is the presence of traces of organic sulfur compounds, CO₂, hydrocarbons, heavy organics, NH₃, and cyanides. Usually these compounds either reduce the catalyst activity by fouling, or dilute the H₂S concentration in the feed, thereby also reducing the sulfur recovery. According to EPA policy on sulfur-emission control, the Claus process alone is not acceptable. To increase the sulfur-removal efficiency from 92-96% (possible with three catalytic stages in Claus process) to about 99.8% (corresponds to about 200-300 ppm in the off-gas), a process for treating tail-gas is required. However, such a process can account for about two-thirds of the cost of the total sulfur-recovery system and a significant portion of the energy requirements.

B. Stretford Process

In this process,^{11a} the sulfur-bearing gas is fed to an absorber where H_2S is reacted as follows:

In the bottom of the absorber, sodium vanadate ($NaVO_3$) oxidizes bisulfide to elemental sulfur as follows:

Vanadium(IV) is reoxidized to vanadium(V) with anthraquinone disulfonic acid (ADA), as follows:

The mixture of sulfur and the solution containing sodium-vanadium salts is mixed with air where ADA is reoxidized:

In the absorber of the Stretford process redwood slats are used to provide good contact of the gases containing H_2S with the Na_2CO_3 solution. Above concentrations of about 5% H_2S , some units are reported to experience plugging with sulfur. Recent developments indicate that a venturi scrubber can be used for feeds above the 5% H_2S level.

Part of the recovered sulfur forms sodium thiosulfate, which is not regenerable and therefore has to be purged from the system to maintain solution activity. Since the cost of the vanadium salts in the Stretford solution is high, this purge must be minimized. As in the Claus process, the performance of the Stretford process is also affected adversely by organic sulfur compounds (such as COS , CS_2), CO_2 , hydrocarbons, and cyanides.

It is claimed by the developer of the process that under ideal conditions the H_2S level in the tail gases can be reduced to 10 ppm with the Stretford process. However, a realistic goal is set around 100 ppm, which is well within EPA limits.

Because the Stretford process does not need a tail-gas clean-up system (as in the Claus process) and is capable of handling H_2S concentrations in the feed material ranging from 5-20%, only the Stretford process for sulfur recovery was considered in the analysis that follows.

V. SEED RECOVERY

Most of the seed (primarily as K_2SO_4) that will be collected in the various downstream components and electrostatic precipitators will be contaminated with some ash. It would obviously be advantageous if, in the seed-regeneration process, small amounts of Al_2O_3 , SiO_2 , Fe_2O_3 , and CaO , etc. mixed with the K_2SO_4 did not affect the conversion to carbonate nor interfere with the combustor and channel operations. However, since none of the candidate processes

have demonstrated this capability, it was assumed in the analyses of the PERC and Tampella processes that separation from most of these insolubles could be achieved by leaching of K_2SO_4 (+ K_2CO_3) with hot water. A schematic drawing of this seed-recovery step is shown in Fig. 1.

Fig. 1. Schematic of Seed-Recovery Process (A, B, C, etc. refer to entries in Table 3.)

According to the basic ground rules, the collected mixture of spent seed and fly-ash would contain 95 wt % K_2SO_4 plus K_2CO_3 , contaminated with 5 wt % fly-ash. Most of the fly-ash portion is assumed to consist of insolubles.

The minimum potassium recovery specified is 95%. The 5% loss of potassium was distributed arbitrarily as follows:

1. 2% lost in MHD plant.
2. 2% lost in seed-recovery step as insoluble and unextractable potassium aluminosilicates (includes inefficiency of extraction, filter loss, etc.).
3. 1% lost in seed-regeneration step (includes inefficiency of regeneration, loss as unconvertible, soluble potassium silicates, etc.).

In the present analysis, it was assumed that a seed recovery step is required for both the Tampella and PERC processes. In the future, it may be possible to avoid a seed recovery step if development work shows that the small amount of ash does not affect recovery of sulfur from K_2SO_4 .

Similarly, in the future, it may be possible that in one or both processes the feed material for the reduction step can be either solid or molten sulfate. However, in the present analysis, the feed was assumed to be a solid mixture of K_2SO_4 and K_2CO_3 containing a small amount (about 10 wt %) of water.

Other important assumptions involved in the analysis of the seed recovery scheme are the following:

1. Because of the high molar ratio of K_2SO_4 to K_2CO_3 in the spent seed, the entire mixture may be assumed to follow the solubility of pure K_2SO_4 .
2. The extraction efficiency of spent seed with hot water is 99%.
3. Thermophysical properties of water were used for the solution and slurry containing potassium salts. Thus, property changes such as elevation in boiling point, differences in heat capacity, etc. were not considered.
4. Various heat losses were neglected.
5. The heat of solution of K_2SO_4 was taken for the case of a very dilute solution.
6. Auxiliary power required to run various pumps and other electrical units was assumed to be about 10% of the total requirement for steam or other forms of energy.

The seed-recovery scheme shown in Fig. 1 and the assumptions outlined above were used to calculate mass and heat balances around major components. The calculated compositions of the various streams, as well as the energy requirements for the major components, are given in Table 3.

In the detailed analyses of seed-regeneration processes described in the following sections, the stream exiting from the seed-recovery hold-up tank served as the input stream for both the PERC and Tampella processes.

Table 3. Flow Chart for Seed-Recovery System

Basis: 1000 Mwt plant

Illinois #6 coal feeding rate = 270,000 lb/hr

Seed feed rate to MHD plant = 46,800 lb/hr (85 wt % K_2CO_3)

Process Stream ^a	Stream Components, lb/hr			Process Water	Steam, lb/hr	Cooling Water, lb/hr	Temp., °F	Enthalpy, 10 ⁶ Btu/hr
	K ₂ SO ₄	K ₂ CO ₃	Ash					
A	52,700	3,600	2,960	--	--	--	60	0.3
B	1,050	72	--	--	--	--	--	--
C	51,600	3,550	2,960	224,000	--	--	212	42.4
D	50,600	3,480	30	224,000	--	--	212	50.1
E	1,050	72	2,930	--	--	--	--	--
F	50,600	3,480	30	6,010	--	--	120	1.5
G	--	--	--	79,400	--	--	120	88.5
^b H ₂	--	--	--	67,800	--	--	227	13.2
^c H ₃ H	--	--	--	71,200	--	--	207	12.5
J	--	--	--	218,000	--	--	182	32.7
K	--	--	--	6,010	--	--	60	0.2
^d L	--	--	--	--	13,400	--	240	15.6
^e M	--	--	--	--	72,800	--	240	84.5
N	--	--	--	--	--	1,360,000	60	38.0

Energy Requirements, Btu/hrHeat required as steam in leaching tank 13×10^6 Heat required as steam in 1st effect of evaporators 69×10^6 Subtotal 82×10^6 Auxiliary power for pumps, filters, etc. (10%) 8×10^6 Total 90×10^6 ^aLetter designations refer to callouts in Fig. 1.^bFrom 2nd effect of evaporators.^cFrom 3rd effect of evaporators.^dSteam pressure of 25 psia.^eSteam pressure of 25 psia.

VI. ANALYSIS OF PERC PROCESS

A seed-regeneration scheme based on the PERC process is shown in Fig. 2. The equilibrium code developed by NASA-Lewis¹⁷ was used to calculate the equilibrium compositions of streams from the gasifier, reducer, and regenerator. The computer program uses the technique of free-energy minimization of the system involving given elements and thus avoids considering only one particular reaction. Thermodynamic properties for K_2S , such as free energy of formation as a function of temperature, were not available; however, they could be

Fig. 2. Seed-Regeneration Scheme Based on the PERC Process (The letters A, B, C, etc. refer to process streams described in Table 4.)

estimated from available thermophysical properties and basic principles of thermodynamics. Some of the values for K_2S that were used are given below:

$$\Delta H_f^\circ, 298 \text{ K} = -99 \text{ kcal/mol (Ref. 18)}$$

$$\Delta S_f^\circ, 298 \text{ K} = -11.5 \text{ cal/mol}\cdot\text{K (estimated)}$$

$$\Delta G_f^\circ, 298 \text{ K} = -95.6 \text{ kcal/mol (estimated)}$$

$$C_p = 16.78 + 4.74 \times 10^{-3}T \text{ cal/mol}\cdot\text{K at } 100 \leq T \leq 700 \text{ K (Ref. 18)}$$

$$C_p = 20.57 \text{ cal/mol}\cdot\text{K at } 800 \text{ K (Ref. 18)}$$

$$C_p = 25.25 \text{ cal/mol}\cdot\text{K at } 900 \text{ K (Ref. 18)}$$

$$C_p = 35.17 \text{ cal/mol}\cdot\text{K at } 1000 \text{ K (Ref. 18)}$$

For simplified calculation in the condensed phase, it was assumed that no interaction or solid solution between different species was possible. In addition, a computer simulation of K_2SO_4 reduction utilizing 50% CO -50% H_2 was made with a modified version of the NASA-Lewis code,¹⁹ which allowed for interaction among the condensed species (such as K_2SO_4 , K_2CO_3 , and K_2S). At 1100 K, with no interaction, 94.8% of the K_2SO_4 was reduced; with the interaction mode, 92% was reduced. This small difference in the percent reduction of K_2SO_4 justified the assumption of no interaction among the condensed species.

In addition to the assumptions made for the seed-recovery scheme, the following rationale was used for analysis of seed regeneration and sulfur recovery:

1. The low-Btu reducing gas for the reducer is supplied by an air-blown-suspension gasifier operated at 2 atm pressure, a pressure considered adequate for transporting material through the system.
2. To obtain 85% conversion of K_2SO_4 to K_2CO_3 with Illinois #6 coal, the gasifier is supplied with an air/coal ratio of 3.5 and a K_2SO_4 /coal ratio of 3.88. Under these conditions at 2 atm pressure, the minimum gasifier temperature at which all the carbon is converted is 1477°C (2691°F). The air to the gasifier is assumed to be preheated to 538°C (1000°F).
3. The temperature of the reducer is 827°C (1521°F).
4. The soluble portion of the ash from the seed recovery scheme consists of Al_2O_3 , SiO_2 , Fe_2O_3 , and CaO , in the same proportion as in the original coal.
5. At a regenerator temperature of 427°C (801°F), complete conversion of K_2S to K_2CO_3 is possible. At this temperature, some 60% of the total off-gases from the reducer is mixed with a small amount of live steam in the regenerator.
6. Only the regenerator off-gases are treated in the Stretford unit for complete sulfur recovery. In the absence of adequate data on the thermodynamics and kinetics of various reaction steps involved in the Stretford process, an overall reaction of

is considered for the mass balance calculations, and no heat balance calculations are made for this step.

7. The 1% loss of potassium that was attributed earlier to the seed-regeneration scheme (due to conversion efficiency and operational loss) is assumed to be incurred equally as K_2SO_4 and K_2CO_3 .

Using these assumptions, and the flow sheet shown in Fig. 2, the compositions and energy contents of the major components were calculated for the various streams. These calculated results are given in Tables 4 and 5.

Table 4. Flow Chart for Scheme Based on PERC Process

Basis: 1000 MWt plant

Illinois #6 coal feeding rate = 270,000 lb/hr

Seed feed rate to MHD plant = 46,800 lb/hr (85 wt % K_2CO_3)

Process Stream ^a	Stream Components, lb/hr					Gas Components, lb-mol/hr								Temp., °F	Enthalpy, 10 ⁶ Btu/hr
	K ₂ SO ₄	K ₂ CO ₃	K ₂ S	Ash ^b	Process Water ^c	Sulfur	CO	CO ₂	H ₂	H ₂ S	O ₂	N ₂			
A	50,600	3,480	--	30	6,010 (1)	--	--	--	--	--	--	--	120	1.5	
B	--	--	--	--	--	--	--	--	--	--	332	1,250	1,000	11.0	
C	(13,000 lb/hr washed Illinois #6 coal)					--	--	--	--	--	--	--	60	164.0	
D	--	--	--	--	2 (g)	--	738	0.1	303	6.0	--	1,250	2,691	47.1	
E ^d	8,690	1,610	28,000	39	--	--	--	--	--	--	--	--	1,521	11.4	
F	--	--	--	--	6,470 (g)	--	30	427	25	1.0	--	753	1,521	30.7	
G	--	--	--	--	4,320 (g)	--	20	285	17	0.9	--	502	1,521	20.5	
H	(1,760 lb/hr live steam @25 psia)					--	--	--	--	--	--	--	240	2.0	
J ^e	7,190	37,800	--	57	--	--	--	--	--	--	--	--	801	6.9	
K	47	2,280	--	--	--	--	--	--	--	--	--	--	60	--	
L	286	227	--	--	--	--	--	--	--	--	--	--	--	--	
M	6,950	39,900	--	--	--	--	--	--	--	--	--	--	60	--	
N	--	--	--	--	3,650 (g)	--	2	192	19	623	--	753	801	13.2	
P	--	--	--	--	4,100 (g)	--	2	193	19	0.1	--	1,250	110	5.4	
Q	--	--	--	--	--	--	--	--	--	--	132	495	60	0.1	
R	--	--	--	--	4,270 (1)	8,430	--	--	--	--	--	--	110	0.5	

^aLetter designations refer to callouts in Fig. 2.^bIncludes compounds such as SiO_2 , Fe_2O_3 , Al_2O_3 , CaO , CaS , FeO , $K_2Si_2O_5$, $CaSiO_3$, FeS_2 , $KAlO_2$, and K_2SiO_3 .^cThe notations (1) and (g) refer, respectively, to liquid and gaseous H_2O .^dAlso contains 4.4 lb/hr KCl , and 10 lb/hr KOH .^eAlso contains 3.9 lb/hr KCl .

Table 5. Overall Energy Balance for Scheme Based on PERC Process

Basis: 1000 Mwt plant
 Illinois #6 coal feeding rate = 270,000 lbs/hr
 Seed feed rate to MHD plant = 46,800 lbs/hr
 (85 wt % K_2CO_3)

	Energy, 10^6 Btu/hr
<u>Required</u>	
As coal in gasifier	164
To preheat gasifier air	11
To maintain reducer temperature	10
As live steam in regenerator	<u>2</u>
Subtotal	187
Auxiliary power (10%)	<u>19</u>
Total required	206
<u>Available</u>	
From reducer off-gases ($1521^\circ F \rightarrow 126^\circ F$)	17
From regenerator (heat of reaction, sensible heat, etc.) (at $801^\circ F$)	39
From regenerator off-gases ($801^\circ F \rightarrow 110^\circ F$)	8
From sensible heat of regenerated seed (at $801^\circ F$)	<u>7</u>
Total available	71
<u>Net Required</u>	
Net = (total required) - (total available)	
= 135×10^6 Btu/hr	

VII. ANALYSIS OF MODIFIED TAMPELLA PROCESS

The products from the seed-recovery step were assumed to follow the scheme shown in Fig. 3 for a modification of the Tampella process based on the use of potassium salts. Because of the lack of information on the thermodynamic properties of KHS, KHCO_3 , etc., the NASA-Lewis computer code¹⁷ could not be used to calculate equilibrium compositions of streams coming out of the pre-carbonator, stripper, and carbonator; therefore, this code was used only for the reaction in the reduction furnace.

The following rationale was used for the analysis and the seed regeneration scheme:

1. For a mixture containing 85% K_2S and 15% K_2SO_4 , the liquidus temperature is about 830°C (1526°F).²⁰ To obtain a final melt of reduced material completely in liquid form, the temperature in the reduction furnace is kept at about 900°C (1650°F).

2. Based on the NASA-Lewis code, adequate reduction of K_2SO_4 (so that the overall conversion from sulfate to carbonate is about 85%) at 1650°F , requires about 17,600 lb/hr of Illinois #6 coal.

3. The reduced molten salts from the reduction furnace are quenched with sufficient water to avoid vapor explosions. As an alternative that eliminates the possibility of explosions and minimizes the amount of water, the melt may be gas-cooled to solidify it, crushed, and then dissolved. (This alternative was not considered in detail.)

4. Because of the low concentration of insoluble materials in the resulting liquor, a filtering step is employed instead of a conventional clarifier to remove insoluble materials such Al_2O_3 , CaS , FeO , $\text{K}_2\text{Si}_2\text{O}_5$, etc.

5. Based on the information from the paper and pulp industry, the pre-carbonator is assumed to operate at 100°F . In the absence of adequate kinetic and thermodynamic data, it is assumed that the reaction

goes to completion. Only one-half of the flue gas from the furnace is utilized for this operation. The other half is assumed to be utilized in the carbonator.

6. The KHCO_3 required to react with KHS in the stripper is obtained by recycling a portion of the K_2CO_3 and reacting it with CO_2 in the carbonator. Because of this recycle of K_2CO_3 , the mass balance computations around the stripper, evaporator-separator, and carbonator are carried out in an iterative manner. The heat requirements of these units are not optimized.

7. For the reaction in the stripper, a limited amount of equilibrium data is available for a batch system involving absorption of H_2S at pressures from 0.02 to 0.7 atm in 2N K_2CO_3 at low temperatures.²¹ Based on the sodium system, the stripper is maintained at 195°F and 0.7 atm total pressure. By extrapolation of equilibrium data to these conditions, it is calculated that for $\text{P}_{\text{H}_2\text{S}}$ varying from 10 to 1 mm Hg, the equilibrium conversion of KHS will change from 42 to 79%. Therefore, it is assumed that in a flowing system involving nonequilibrium conditions, all of the KHS will be converted to H_2S .

Fig. 3. Seed-Regeneration Scheme Based on Modified Tampella Process (the letters A, B, C, etc. refer to process streams described in Table 6)

8. To economize on steam consumption, a three-effects evaporator system is used. The third effect can very well be a combined crystallizer-evaporator system. Options for separation in this component include a settling tank, a centrifuge, and a filter.

9. From Bureau of Mines' work²² on the equilibrium in a K_2CO_3 - H_2O - CO_2 - $KHCO_3$ system, it is calculated that in a batch system, at 100°F, an equilibrium conversion of about 81% is possible. Since the carbonator can be a reaction tank containing slurry/solution of K_2CO_3 with a continuous sparge of the flue gases containing CO_2 , it is assumed that about 80% of the K_2CO_3 introduced in the combustor will be converted to $KHCO_3$.

10. As in the PERC process, a Stretford unit is selected to recover sulfur. In the absence of adequate information, rigorous analysis around the Stretford unit is not possible. Also, the 1% loss of potassium that was attributed to the seed regeneration scheme (due to conversion efficiency or operating loss, etc.) is assumed to be incurred equally as K_2SO_4 and K_2CO_3 .

Based on these assumptions and the flow sheet shown in Fig. 3, compositions and energy contents of the major components were calculated for the various streams. These results are given in Tables 6 and 7.

Table 6. Flow Chart for Scheme Based on Modified Tampella Process

Basis: 1000 Mwt plant

Illinois #6 coal feeding rate = 270,000 lb/hr

Seed feed rate to MHD plant = 46,800 lb/hr (85 wt % K_2CO_3)

Process Stream ^a	Stream Components, lb/hr						Process Water ^c	KCl, KOH	Gas Components, lb·mol/hr						Temp., °F	Enthalpy, 10 ⁶ Btu/hr
	K_2SO_4	K_2CO_3	K_2S	$KHCO_3$	KHS	Ash ^b			CO	CO ₂	H ₂	H ₂ S	O ₂	N ₂		
A	--	--	--	--	--	--	--	--	--	--	--	--	557	2,090	600	11.3
B	50,600	3,480	--	--	--	30	6,010 (l)	--	--	--	--	--	--	--	120	1.5
C	(17,600 lb/hr washed Illinois #6 coal)					--	--	--	--	--	--	--	--	--	60	222.0
D	--	--	--	--	--	--	12,600 (g)	--	82	954	48	4	--	2,100	1,650	81.3
E	3,760	--	31,400	--	--	2,800	--	47	--	--	--	--	--	--	1,650	12.3
F	--	--	--	--	--	--	294,000 (l)	--	--	--	--	--	--	--	60	8.2
G	3,760	--	31,400	--	--	2,800	294,000 (l)	47	--	--	--	--	--	--	100	20.5
H	--	--	--	--	--	2,800	140 (l)	--	--	--	--	--	--	--	100	--
J	3,760	--	31,400	--	--	--	294,000 (l)	47	--	--	--	--	--	--	100	20.5
L, M	--	--	--	--	--	--	1,980 (g)	--	41	477	24	2	--	1,050	100	3.1
N	3,760	19,730	--	--	20,560	--	292,000 (l)	6	--	--	--	--	--	--	100	20.4
P	--	--	--	--	--	--	55,900 (g)	--	--	--	--	285	--	--	195	64.4
Q	--	--	--	--	--	--	55,200 (l)	--	--	--	--	--	--	--	110	4.3
R	--	--	--	--	--	--	725 (g)	--	--	--	--	285	--	--	110	1.0
S	--	--	--	--	--	--	--	--	--	--	--	--	143	536	60	0.1
T	--	--	--	--	--	--	917 (g)	--	--	--	--	0.1	--	536	110	1.3
U	(9,125 lb/hr sulfur in form of water slurry)					--	--	--	--	--	--	--	--	--	110	0.2
V	(94,000 lb/hr steam at 14.7 psia)					--	--	--	--	--	--	--	--	--	212	108.0
W	(80,600 lb/hr steam at 25 psia)					--	--	--	--	--	--	--	--	--	240	93.6
X	5,260	64,000	--	--	--	--	241,000 (l)	9	--	--	--	--	--	--	195	41.5
Y	5,260	64,000	--	--	--	--	7,530 (l)	9	--	--	--	--	--	--	100	1.4
Z	--	8,730	--	--	--	--	7,530 (l)	--	--	--	--	--	--	--	100	0.6
a	1,500	15,900	--	--	--	--	--	3	--	--	--	--	--	--	100	0.2
b	1,500	4,920	--	28,500	--	--	5,140 (l)	3	--	--	--	--	--	--	100	0.8
c	286	227	--	--	--	--	--	--	--	--	--	--	--	--	--	--
d	3,480	719	--	--	--	--	--	--	--	--	--	--	--	--	60	--
e	6,950	39,900	--	--	--	--	--	6	--	--	--	--	--	--	60	--

^aLetter designations refer to callouts in Fig 3.^bIncludes compounds such as SiO_2 , Fe_2O_3 , Al_2O_3 , CaO , CaS , FeO , and $K_2Si_2O_5$.^cThe notations (l) and (g) refer, respectively, to liquid and gaseous H_2O .

Table 7. Overall Energy Balance for Scheme Based on Modified Tampella Process

Basis: 1000 Mwt plant
 Illinois #6 coal feeding rate = 270,000 lbs/hr
 Seed feed rate to MHD plant = 46,800 lbs/hr (85 wt % K_2CO_3)

	Energy, 10 ⁶ Btu/hr
<u>Required</u>	
As coal in reduction furnace	222
To preheat furnace air	11
In stripper as steam	91
As steam in evaporator (1st effect)	<u>77</u>
Subtotal	401
Auxiliary power (10%)	<u>40</u>
Total required	441
<u>Available^a</u>	
From quenching of melt (1650°F → 100°F)	12
From furnace off-gases (1650°F → 100°F)	<u>75</u>
Total available	87
<u>Net Required</u>	
Net = (total required) - (total available)	
= 354 × 10 ⁶ Btu/hr	

^a Low-temperature waste heat available from the precarbonator, evaporator, carbonator, regenerated seed, and gaseous effluent from stripper are not considered here.

VIII. COMPARISON OF ENERGY REQUIREMENTS FOR PERC AND TAMPELLA PROCESSES

In Table 8, total and net energy requirements for the PERC and Tampella processes are compared with similar results available from the literature. In Table 9, comparison is made between the calculated energy requirements for the Tampella process, as modified for the potassium system, and the actual energy requirements for the sodium system published by the paper and pulp industry.⁵

From Table 8, it is clear that the PERC process has lower energy requirements per pound of potassium than the Tampella process. The higher energy requirements in the latter process can be attributed to higher consumption of coal (for reducing purposes), and the required evaporation of water.

Table 8 illustrates the variations in calculated energy requirements that are obtained¹² even for a single process (the PERC process), when different values are assumed for gasifier efficiency and gas utilization.

The comparison made in Table 9 between the calculated energy requirements for the modified Tampella process (for the potassium system) and the energy actually used for the sodium system in industry shows a satisfactory correlation. The difference that is seen can be attributed to (1) differences in the kinetics and thermodynamics of the potassium and sodium systems and (2) the nonoptimized heat-balance computation in the present analysis.

Energy requirements for the Formate and AC processes are also listed in Table 8. The Formate process has not yet been ruled out by the MHD community and the AC process (which is described in the AVCO report as the Carbon Reduction process) is very similar to the Tampella process; hence, some consideration of these processes is warranted.

In the Formate process described in the AVCO report⁹ a seed-recovery step was not employed and the seed-regeneration scheme was considered only up to the production of potassium formate (instead of the more conventional K_2CO_3). Because of this modification, the AVCO report claims that the scheme will have a low energy requirement. From Table 8 it can be seen that their statement is correct from the standpoint of net energy requirement but not from the total energy requirement. AVCO's⁹ calculations for the Carbon Reduction process (Aqueous Carbonate process) show a lower net energy requirement than the Tampella process considered here. Their lower net energy for the AC process is possible for two major reasons. The first is that AVCO considered about 40 wt % solution of potassium salts in the quench tank vs. 11 wt % solution used in this analysis. Because of this, their energy requirement for removal of water has been greatly reduced. (In the paper and pulp industry, for the black liquor recovery process, a 10 wt % solution is used for the smelt quenching system.⁹) The second reason is that AVCO also gave credit for the thermal energy available in the reduction furnace off-gases, which contained some CO.

Table 8. Comparison of Energy Requirements per Hour for Various Seed-Reprocessing Schemes (for Illinois Coal)

Scheme	Plant Size, MWt	Potassium Handled, lb	Energy Required, Btu/lb K	
			Net	Total ^a
<u>This study</u>				
Seed recovery	1,000	25,200	--	3,570
PERC process	1,000	24,600	5,490	8,370
Tampella process	1,000	24,600	14,400	17,900
<u>From literature:</u>				
PERC process ^b	2,000	32,900	4,790	10,900
PERC process ^c	730	--	9,710	--
PERC process ^d	730	--	5,740	--
Carbon reduction process ^e	280	4,450	5,980	23,900
Formate process ^f	190	4,280	2,560	11,400

^aDoes not include the credit for available waste-heat.

^bFrom Ref 23. Does not include cleanup of Claus plant tail gases.

^cFrom Ref. 12. Based on 90% thermal efficiency for gasifier, and 21% gas utilization. Amount of potassium handled cannot be estimated.

^dFrom Ref. 12. Based on 85% gas utilization. Amount of potassium handled cannot be estimated.

^eFrom Ref. 9. Does not include H₂S removal step.

^fFrom Ref. 9. Considers only up to the formation of potassium formate from K₂SO₄.

Table 9. Comparison of Industrial Energy Requirements for Tampella Process with Requirements Calculated in This Study

Source	Quantity of H ₂ S Produced, lb·mol/hr	Energy Consumption (on H ₂ S Basis), Btu/lb·mol
Kemi Oy (Finland) ^a	43	1.5×10^5
Alizay (France) ^b	22.5	5.6×10^5
This study ^c	285	Net, $\sim 12 \times 10^5$ Total, $\sim 15 \times 10^5$

^aReported as actual heat consumption for the sodium system (Ref. 5).

^bReported as actual heat consumption for the sodium system (Ref. 5).

^cNet and total energy requirements are given for potassium system.

IX. ECONOMIC COMPARISON

As discussed earlier, an economic analysis was not attempted in this work; however, results of economic analyses available from the literature are summarized in Table 10.

The reports from Westinghouse¹² and PERC^{23,24} used different bases to describe their respective plant sizes. For one of the PERC reports,²³ handling capacity in terms of pounds of K_2SO_4 per hour was estimated from the reported mass balance. Accordingly, as shown in the PERC calculations, \$28.8 million was required as a total capital cost vs. \$37.3 million required as a total capital cost by Westinghouse.¹² When the difference in plant size is considered, the agreement in capital cost is considered reasonable. A previous cost estimate of the PERC process,²⁴ based on regeneration of seed as KOH, rather than K_2CO_3 , was \$10.4 million. The increase to \$28.8 million²³ resulted from the additional high-temperature step required for conversion of K_2S to K_2CO_3 .

Also listed in Table 10, are the available cost figures for the Double Alkali process, the Aqueous Carbonate process, and a sulfur throw-away process.

In the Westinghouse analysis,¹² it is clear that the capital cost for the Double Alkali process is lower than either the PERC or the AC process. However, the requirement of very dilute solutions to achieve the necessary conversion results in a subsequent, large increase in load in the evaporation step; therefore, the operating cost for the Double Alkali process would be quite high. The operating cost of the potassium throw-away alternative is unacceptably high because of the replenishment cost for the seed (the cost of K_2CO_3 is about \$420/ton). The AC process, as reviewed in Ref. 12 had a high capital cost, probably because of its requirement of a two-compartment molten-salt reactor. However, in the revised version^{15a} of the AC process only a single compartment reactor is used; therefore the capital cost may be somewhat lower. For the PERC process, the reasons for high capital cost were attributed to the requirements of a coal gasifier and a gas-handling section.

The AVCO report⁹ also compares the cost of a seed regeneration process with available flue-gas desulfurization (FGD) processes. For a 250-MWt plant fueled with low-sulfur Montana coal, the capital cost for a limestone slurry/flue gas desulfurization system is estimated to be double that of the Formate process.

Table 10. Economic Comparison of Various Seed-Reprocessing Schemes

Scheme	Plant Size		Estimated Capital Cost 10 ⁶ \$	Estimated Annual Cost, 10 ⁶ \$ ^a	Source
	Power Rating, MWt	K ₂ SO ₄ Handling Capacity, lb/hr			
PERC process	--	79,200	37.3	2.95	Westinghouse, Ref. 12
PERC process	--	15,840	--	0.84	
PERC process ^b	2,000	73,400	28.8	--	PERC, Ref. 23
PERC process ^c	2,000	--	7.56	3.69	PERC, Ref. 24
Seed recovery ^c	2,000	--	1.77	2.33	
Double Alkali process	--	79,200	7.12	2.99	Westinghouse, Ref. 12
Double Alkali process	--	15,840	--	0.62	
Aqueous Carbonate process	--	79,200	44.1	2.23	
Aqueous Carbonate process	--	15,840	--	0.59	
Throw-Away process	--	79,200	--	70.00	
Throw-Away process	--	15,840	--	14.00	

^aIncludes operating and maintenance cost.

^bSeed recovery and regeneration are included together. Capital cost is given as total capital investment (interest, installation cost, depreciation, etc. are included); K₂SO₄ handling capacity is estimated. Regenerated seed is in the form of K₂CO₃.

^cTotal capital cost is given as $\$10.4 \times 10^6$; regenerated seed is in the form of KOH.

X. CONCLUSIONS

Under the conditions and assumptions of this evaluation study, the PERC process appears to have lower energy requirements per pound of potassium handled than the modified Tampella process. However, gas utilization in the PERC process needs to be improved. Although the Tampella process shows higher energy requirements (gross as well as net), it has the distinct advantage that most of its components have been demonstrated successfully, with the sodium system, in the paper and pulp industry. With better furnace design and a different method of quenching the melt, it may be possible to reduce the energy requirements for the Tampella process. Because the Formate process did not meet the requirements imposed as the bases of this evaluation, the Formate process was not analyzed in any detail; however, the work at AVCO⁹ indicates that energy requirements for the Formate process are quite moderate. Moreover, the capabilities of the process have been demonstrated in industry.

The capital cost for the PERC process appears to be lower than that for the AC process. The conceptual designs of the Tampella and the AC processes do not differ significantly; thus, it may be assumed that the capital cost for the Tampella process will be somewhat lower than the AC process because the Tampella process does not need a two-compartment molten-salt reactor that appears to have been used in the economic analysis of the AC process. With assumption of a single molten-salt reactor in the Tampella process, the difference in the capital cost between the PERC and Tampella processes should be small. Similarly, it can be concluded that the difference in the annual operating cost between the PERC and Tampella processes will be small. Thus, based on economic considerations, there may not be any significant difference between the PERC and Tampella processes.

Another important factor which must not be overlooked is potassium-ash complexing in the AC and Tampella processes. Low-ash petroleum coke could, perhaps, be used in both processes but the operating cost would be higher than with Illinois coal. In this work, it was calculated that, in addition to the 5% loss that was assumed, a further potassium loss of about 3% would be incurred in the modified Tampella process. Another potential disadvantage for the AC, Tampella and probably PERC processes is the materials problem. The furnace used by Atomics International for the AC process has walls constructed of fused alumina; AI claims that this material withstands the attack of molten sodium salts. However, its resistance to molten potassium salts is uncertain.

XI. RECOMMENDATIONS

It should be understood that the designs presented herein are preliminary concepts based on theoretical analysis. Application of these concepts directly to engineering design is presented only in a preliminary way. The technical uncertainties inherent in this kind of analysis can be resolved only by additional experimental work either on a bench scale or on a pilot-plant scale. Since none of the processes has a distinct advantage over the others, it is recommended that, if possible, experimental work be carried out for the Modified Tampella as well as the PERC process. More specifically, additional developmental work is recommended in the following areas.

A. Modified Tampella Process

1. The critical component in the modified Tampella process is the reduction furnace. To achieve maximum reduction of K_2SO_4 with a minimum amount of coal and a minimum loss of potassium with the ash components, this unit needs the most developmental work. Information such as kinetic rate data, compatibility of construction materials with molten potassium salts, amounts of potassium lost as a function of type of carbon-containing materials, temperature, etc. are also needed to design an efficient furnace.

2. Quenching of the melt is another process step which needs more work. Additional experimental work is needed to determine the conditions and concentrations under which vapor explosions can be prevented. Atomics International⁴ claims that as an alternative to water-quenching of the melt, the melt can be gas-cooled, solidified, and then broken into chunks for dissolution. Since the solidified melt is quite porous, they think that it will dissolve quickly without any trouble in a lesser amount of water than required for direct water quenching of the melt.

3. Experimental support is required for the efficient design of the precarbonator, stripper, and carbonator. The information that is available at present is for a batch system at lower temperatures. Kinetic data for a flowing system for each of these components in the presence of soluble ash compounds and other potassium salts will be quite helpful. Also, the alternative scheme of combining the stripper and carbonator, as in the Aqueous Carbonate process, should be studied in detail to evaluate its impact on H_2S concentration, energy consumption, etc.

B. PERC Process

1. Critical components for the PERC process are the coal gasifier, reducer, and regenerator. All three need to be developed simultaneously, because the effectiveness of one is reflected in the performance of the others. Kinetic rate data need to be obtained for reducing agents such as mixtures of CO and H_2 , synthesis gas, producer gas, coal, etc. Experiments should be carried out, if possible on a pilot-plant scale, simulating stagewise operation or different reactor geometries with gas recycle to improve gas utilization.

2. In the reduction and carbonation steps, reaction-controlled mechanisms (as yet unidentified) have been suggested as the possible reasons for poor gas utilization in the PERC process, rather than mass transfer or gaseous diffusion-controlled mechanisms.⁹ If this is true, then catalytic reaction or reaction under slight gas pressure might prove advantageous. In other words, reduction and regeneration steps of the PERC process should be examined under MHD environments with the aim of understanding the basic reaction mechanisms. In this way, poor gas utilization and low overall reaction rates might be eliminated. Some of the ash compounds are reported to show catalytic activity, and their use as catalysts may simplify or eliminate the seed-recovery step, which is aimed specifically at separation of potassium from the ash compounds.

C. General

In addition to the specific areas outlined above, other common areas where additional work is required are as follows:

1. Thermodynamic data on compounds such as K_2S , KHS , $KHCO_3$, etc. are either inadequate or unreliable. To carry out effective equilibrium analyses, reliable values for thermodynamic properties are needed.

2. Materials problems resulting from the use of potassium salts, either molten or in aqueous solution under varying temperature conditions, should be given a high priority. Both erosion and corrosion problems need to be addressed. Helpful information may be obtained from the planned 100-MW demonstration plant at the Huntley station in which the AC process (based on a sodium system) will be used. However, a great number of extrapolations will have to be made in going from a sodium system to a potassium system.

3. In the regenerated seed, a significant amount of chloride (which comes from the coal) will be present as KCl in the Tampella process and to a lesser extent in the PERC process. It is suspected that the chloride ions may have strong adverse effects on the electrodes as well as the refractories. Ways of removing chloride must be studied.

4. The low energy requirement for the Formate process estimated by AVCO⁹ and the use of potassium formate as a seed material in the MHD combustor should be verified. If the guidelines for sulfur recovery are changed to allow for throw-away sulfur compounds (such as $CaSO_4 \cdot 2H_2O$), then the Formate process is certainly attractive. Moreover, it has the advantage of being a proven industrial process.

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APPENDIX.

Unit Conversion Factors

To convert from	To	Multiply by
lb/hr	kg/s	1.26×10^{-4}
°C	K	$t_K = t_C + 273.15$
°F	K	$t_K = (t_F + 459.67)/1.8$
Btu/hr	W	2.933×10^{-1}
lb/in. ²	Pa	6.895×10^3
cal	J	4.19
Btu/lb	J/kg	2.328×10^3
lb·mol/hr	kg·mol/s	1.26×10^{-4}

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