LOW-TEMPERATURE MICROWAVE PYROLYSIS OF SEWAGE SLUDGE

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ABSTRACT

Microwave pyrolysis is proposed as one of several optional technologies for disposing and recycling sewage waste in Malaysia. In this study, sewage sludge was dried and pyrolyzed at low temperature (maximum 650ºC) in a single process at laboratory scale. Sewage sludge was placed in a quartz reactor, which was placed in a microwave cavity oven. The modified household microwave oven used has a frequency of 2.45 GHz and input power of 700 W. Graphite was used as microwave absorber in order to facilitate the sewage sludge to reach temperature required for pyrolysis process to take place. The carbonaceous residue (char) and pyrolytic oil produced were analyzed for the proximate and ultimate composition and the gross calorific value. It is found that in this study, the overall heating rate was 118 ºC/min with heating time of 5 minutes. Microwave pyrolysis of sewage sludge at 650°C gives rise to formation of about 28% char, 6% pyrolytic oil and 68% non-condensable gases (dry basis).The gross calorific value of the pyrolytic oil was 28852 kJ/kg, which is higher than that of lignite and sub-bituminous coal thereby reflecting the potential of this fraction as fuel material.

Keywords: *sewage sludge, pyrolysis, low-temperature, microwave, drying*

INTRODUCTION

The problem of sewage sludge disposal is proving to be one of the most complex environmental problems nowadays. The amount of sewage sludge generated by wastewater treatment plants has been increasing at a rapid pace in recent years and has drawn serious attention from the society. In Malaysia, approximately 3 million m^3 of sewage sludge is produced by Indah Water Konsortium (IWK) annually and the total cost of managing was estimated at RM 1 billion. This sludge volume is expected to rise to 7 million m^3 by year 2020 [1].

Handling this waste is not easy and inevitably gives rise to some collateral pollution. Present practice in Malaysia is either to co-dispose it with solid waste at landfill sites or direct disposal in shallow trenches [1]. However, disposal by land filling and trenches require a lot of space and the soil has to be sealed adequately to prevent leaching of harmful compounds. Therefore, the country has to adopt a more practical, economic and acceptable approach in managing and disposing sewage sludge.

Sewage sludge is abundant in volatile matter and therefore represents a valuable resource which can be converted to useful products if it is subjected to the suitable treatment. In the past decade, the pyrolysis of sewage sludge is receiving increasing attention as an economic and environmentally acceptable route to waste disposal. The products of pyrolysis are gas, oil and carbonaceous residue. More importantly, the gas can be used as fuel [2,3,4]. The carbonaceous residue can also be burnt as fuel, disposed of – since the heavy metals are fixed inside the carbonaceous matrix – or be upgraded to activated carbon [5], and the oil can either be used as fuel or as raw material for chemicals [6].

In microwave pyrolysis, sewage sludge with high moisture content undergoes drying and pyrolysis in a single step [3, 5]. An advantage in microwave process is the short time needed to achieve heating compared to conventional heating methods [7]. Other characteristics of microwave process that are not available in conventional processing of materials are; penetrating radiation, controllable electric field distributions, selective heating of materials through differential absorption, and self-limiting reactions [8].

In previous studies conducted on microwave pyrolysis of sewage sludge, focused was the sewage sludge pyrolysis at high temperature using a 1000 W microwave oven, (temperature ranging from 800 to 1000°C) which maximizes the production of non-condensable gases [3, 5, 7]. Although thorough discussions on the compounds present in the pyrolytic oil produced were made [5, 7] there has been no published research that investigates the microwave pyrolysis of sewage sludge at moderate temperature, using a lower input power microwave oven, so far. The present study provides information on microwave pyrolysis of sewage sludge at lower temperature (650°C), using a modified microwave oven with lower input power (700 W). Two techniques: pyrolysis and microwave heating were applied in this study. An effort was made to take advantage of both the significantly volume reduction together with the production of valuable gases and oils afforded by pyrolysis, and rapid heating that can be achieved with a microwave oven [3].

MATERIAL

Aerobically digested sewage sludge was in used in this study to investigate the effects of microwave pyrolysis temperature on products distribution. Table 1 shows the analyses of the sewage sludge sample. The sewage sludge used in this study had a moisture content of about 80 wt% of the total weight of the sludge. The high moisture content makes that an important amount of water has to be dried off from the sample before the start of pyrolysis. The ash content of the sludge sample was 30.83 wt%, indicating the amount of inorganic matter present in the sewage sludge.

Table 1: Proximate and ultimate analyses of the aerobically digested Klang Valley, Malaysian sewage sludge

Proximate analysis (wt%)	
Moisture (as fed)	80.41
Ash (dry basis)	30.83
Volatile matter (dry and ash free basis)	54.70
Fixed carbon (dry and ash free basis)	14.47
Ultimate analysis (wt%, dry and ash free basis)	
C	33.79
H	5.35
N	5.74
S	0.93
O (by difference)	54.18
Calorific value (kJ/kg, dry basis)	12 365

Due to aerobic treatment received by the sewage sludge in the wastewater treatment plant, the volatile matter content of the sludge is quite significant. Nearly 55 wt% of the dry sludge sample corresponded to the organic fraction which makes this sludge an interesting source of organic compounds when they are subjected to pyrolysis. Fixed carbon, the amount of carbonaceous residue less the ash remaining in the test container after the volatile matter has been driven off in making the proximate analysis of the dry sewage sludge sample, was calculated as 14.47 %.

The most abundant element in the organic fraction is oxygen with value more than 50 % and nitrogen represents the smallest amount (5.75 %).The organic fraction contains 33.79 % carbon and only 5.35 % hydrogen. Thus, the H/C atomic ratio is given as 1.89. This value is higher than those found for other wastes such as wood and rice husks with values near to 1.4, and similar to the H/C values of biomass. The high value obtained for the H/C atomic ratio is an indicative of the strong aliphatic character of the sludge, suggesting the presence of long chains with CH_2 groups [5].

EXPERIMENTAL

For the microwave pyrolysis experiments, wet sewage sludge blended with 5 wt% of graphite was used as the feed material. Powder form of graphite was used because it is easily available. Besides, it provides uniform heating and avoids hot spots at the beginning of the heating process.

The sewage sludge samples were placed in the quartz reactor, which was placed inside the microwave cavity. The reactor is 50 mm diameter x 200 mm length, constructed of quartz with a 13 mm inner diameter of gas inlet and outlet. The input power of the microwave equipment was set at 700 W and the microwave frequency used was 2.45 MHz. An infra red thermometer was used for monitoring the bed temperature. Helium was used to create an inert atmosphere in the reactor. The purge gas outlet located above the heated zone was connected to a condenser filled with dichloromethane. The cooling medium for the condenser is tap water. The other end of the condenser was connected to the gas probe of the MRU Air Fair Emission Monitoring Systems: Exhaust Gas Analyzer DELTA 1600L. Figure 1 shows the schematic diagram of the experimental apparatus.

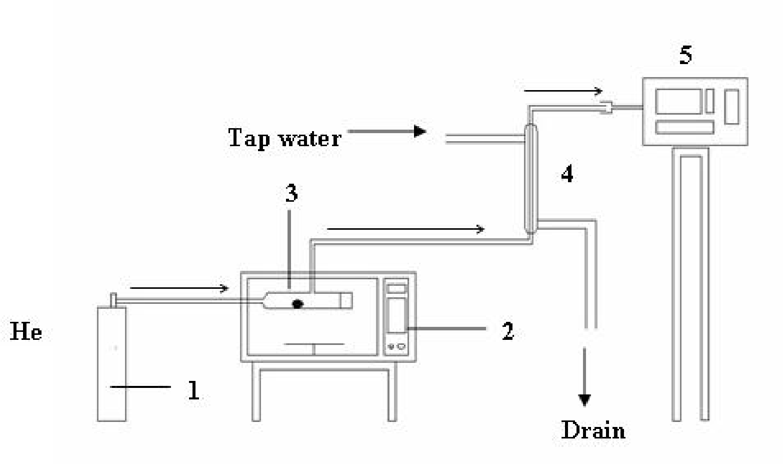

Figure 1: Schematic diagram of fluidized bed pyrolysis of sewage sludge system.
1) Helium gas tank, 2) microwave oven, 3) quartz reactor, 4) condenser, and 5) gas analyzer.

In the experiment, sewage sludge sample was dried and pyrolyzed in a single process using a microwave oven. The prepared samples of 30 g were placed in the quartz reactor. The samples were then subjected to microwave action for about 1, 2, 3, 4, 5 and 6 minutes. In order to maintain an inert atmosphere during the treatments, a helium flow rate of 100 ml/min was passed through the sample bed for 10 minutes prior to the commencement and during the experiments. The process was terminated automatically when the timer set on the control panel of the microwave stopped. Material balances were taken of sludge consumed and all products collected. The carbonaceous residue (char) was easily recovered directly from the quartz reactor. Liquid products were retrieved from the apparatus with dichloromethane [5, 7]. The condenser, tubes, and other equipments where oil may have deposited were washed with dichloromethane as soon as the experiment was finished to recover the maximum amount of volatile released.

The temperature of the sample during the experiments was monitored by the Raytek Raynger ST80 infra red thermometer. Accurate measurement of temperature evolution during the process was very difficult: firstly, there are inherent difficulties involved in measuring temperature in microwave devices; secondly, some of the volatiles evolved during the pyrolysis may condense on "cold" zones of the walls of the quartz reactor making it dim and so filtering the radiation produced by the sample, all of which obstructs measurement with an infra red thermometer. Finally, the temperature, especially at the beginning of the pyrolysis process, is not uniform throughout the sample due to arching, which gives rise to hot spots [3, 5]. Despite these difficulties temperature evolution was followed by means of various experiments carried at 1, 2, 3, 4, 5 and 6 minutes of microwave treatment.

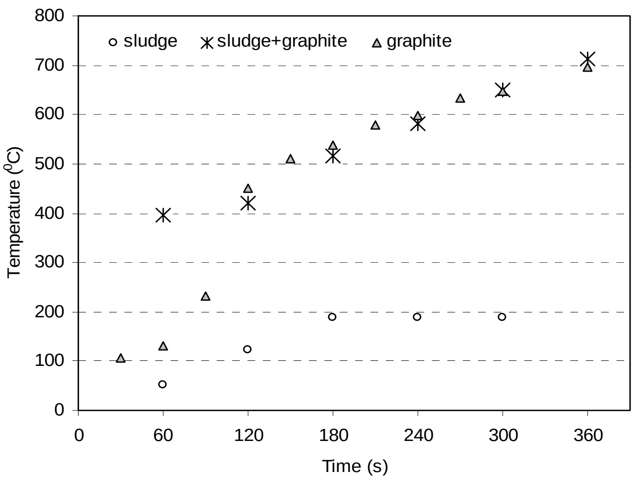

Figure 2: Temperature profile during microwave treatment of graphite, sewage sludge as received;and sewage sludge mixed with 5 wt% of graphite.

RESULTS AND DISCUSSIONS

Figure 2 shows the effect of heating time on the sewage sludge temperature during microwave pyrolysis using microwave oven with 700 W input power.

Microwave treatment of the sewage sludge mixed with 5 wt% graphite results in a maximum bed temperature of about 650 °C after 5 minutes treatment. This is particularly high compared to temperature of 188 °C reached in the same duration of sewage sludge alone. Minimum temperature required in order for pyrolysis to take place is 250 °C [9]. The results obtained indicate that treating the sewage sludge alone cannot achieve the temperature necessary to complete pyrolysis by means of microwave heating. Microwave treatment of the wet sewage sludge (as received) in an inert atmosphere gives rise only to sludge drying. In fact, sewage sludge is a poor receptor of microwave energy with which it is impossible to achieve the temperature necessary to complete pyrolysis [5]. The results show that by adding a small amount of graphite to the sewage sludge sample, microwave pyrolysis of sewage sludge is made possible.

The changes in yield percentage with pyrolysis temperature are shown in Figure 3. The yields for char and oil are calculated on the dry basis, while the gas fraction is calculated by difference. During this treatment the production of char decreases with increasing temperature and heating time. On the other hand, the production of oil and gas increases with the raise in temperature.

As heating time was prolonged, the amount of char gradually decreased. Significant decrease in the char yield occurred during the first 3 minutes of treatment, where the average heating rate was about 170ºC/min. During this period, the bed temperature rise from 397 to 517ºC. The char yield decreased slowly between 580 and 650°C (between the third and fifth minutes). After that, the mass of char yield remained constant. In conclusion, microwave pyrolysis of sewage sludge at 650°C by using the 700 W power input yields about 27.7 wt% of char at the end of the treatment. In fact, high temperature and fast heating rates were determined to decrease the yield of char [10]. This decrease is likely due to the destruction of the organic composition of the sludge and volatiles release that contributed to the formation of pyrolytic gases and oil.

The pyrolytic oil yield is directly proportional to the bed temperature in the microwave pyrolysis treatment. During the microwave treatment sludge sample was heated directly, so it reaches a high temperature in a very short time while the reactor walls remain at lower temperature than the bulk sample. Under these conditions the residence time of the volatiles in the hot zones is relatively short, which does not favour secondary reactions [3]. For this reason no decrease in oil yield was observed during the treatment. At lower temperature, the oil yield was very low. Even at the bed temperature of almost 400°C no oil was produced, but, nevertheless increased gradually with reaction temperature. This condition is not typical in the conventional pyrolysis of sewage sludge where oil may be produced even at lower pyrolysis temperature. The probable reason is most of the microwave energy is used to remove water from the highly moistured sludge at lower temperatures. The conversion process begins only at temperature of about 425°C, where the oil production increases significantly to 5.62% at 650°C.

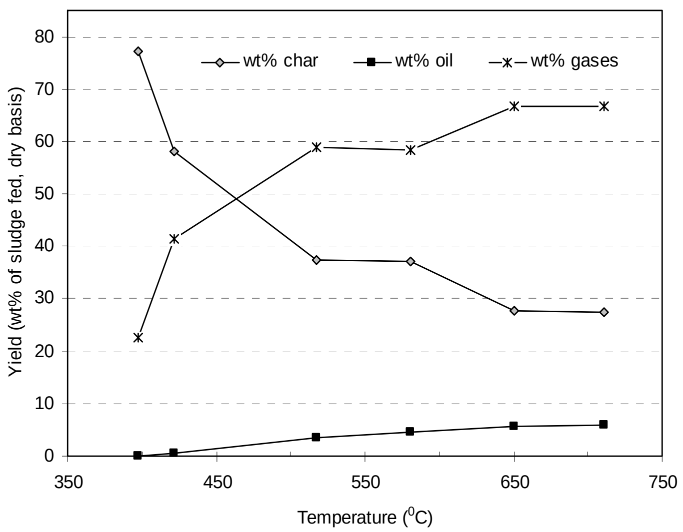

Figure 3: Effects of temperature of the yields of different fractions in microwave pyrolysis of the sewage sludge

Ultimate analyses of the pyrolytic oil produced in the microwave pyrolysis of sewage sludge at 650°C are summarized in Table 2. It was found that the pyrolytic oil had a lower oxygen content and a higher H/O atomic ratio than the initial sludge. The oil is, therefore, considerably deoxygenated so that a large number of functional groups must have been lost during the pyrolysis [7]. The pyrolytic oil had higher carbon content than the initial sludge. The sulphur content of the pyrolytic oil is slightly higher than the maximum sulphur limit permitted by the US EPA where 0.05 wt% (500 ppm) of sulphur is permitted for non-road diesel fuel. The H/C atomic ratio of the oil suggests the presence of compounds with a high aliphatic content [7]. Nevertheless, the H/C value is lower than those for the sludge, which indicates that some aromatisation reactions must have occurred.

Table 2: Ultimate analysis and calorific value of pyrolytic oil produced in low temperature microwave pyrolysis of sewage sludge

Ultimate analysis (wt%, dry and ash free basis)	
C	52.52
H	6.57
N	1.27
S	0.56
O (by difference)	39.09
Calorific value (kJ/kg, dry basis)	28 852

Figure 4 and 5 summarize the calorific value (CV) of the pyrolytic oil obtained in the microwave pyrolysis experiment in comparison to the CVs of the initial sludge sample, and in comparison to other types of fuel. Results from the present study were compared to previous studies. It was found that the CV of the pyrolytic oil obtained in microwave pyrolysis treatment by using microwave oven with 700 W power input (labelled as oil A) at 650°C was 28852 kJ/kg, about double that of the initial sludge. This value is higher than the pyrolytic oil produced at the same temperature by conventional pyrolysis of sewage sludge.

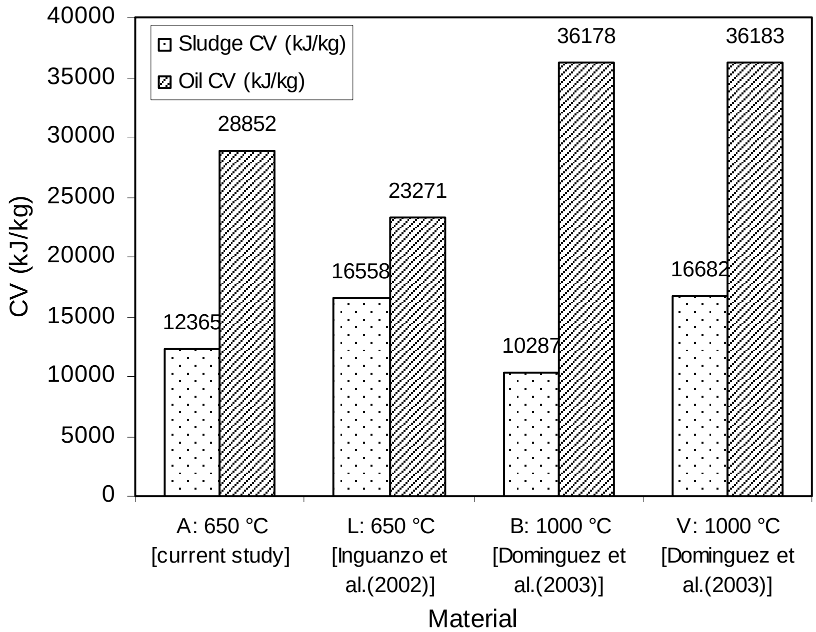

Figure 4: CV of sewage sludge and their pyrolytic oils. A, B and V were subjected microwave pyrolysis. L undergoes conventional pyrolysis at 650 °C

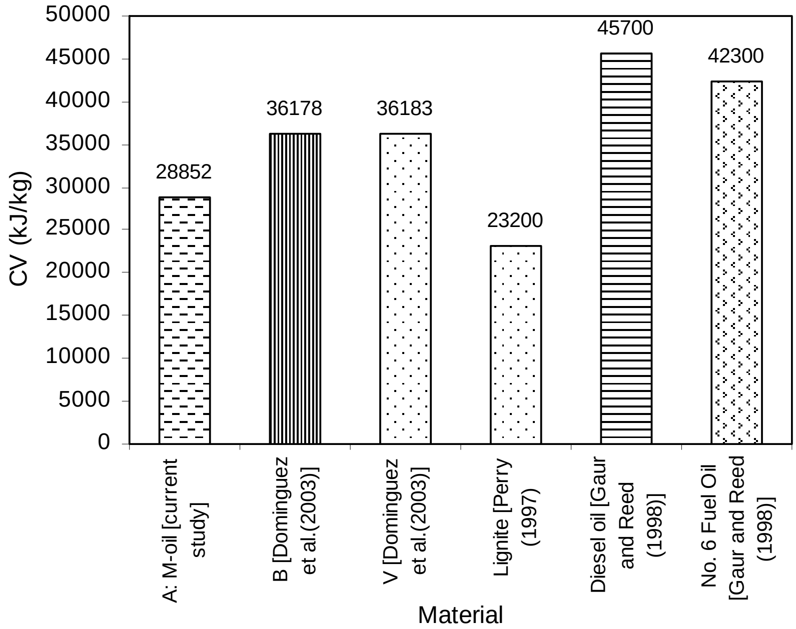

Figure 5: CVs of microwave pyrolytic oils A, B and V in comparison with several fuel materials.

However, the CV of pyrolytic oil produced in the current study is somehow lower than oil B and V. These are pyrolytic oils produced by sewage sludge samples subjected to the same treatment but using a microwave oven with 1000 W input power with maximum temperature of 1000 °C [5, 7]. The CV ranges between 36178 and 36183 kJ/kg. In this case, initial CV for the sludge samples were 10287 and 16682 kJ/kg respectively. It can be implied that at temperature difference of 350°C, additional CV of 7326 to 7331 kJ/kg were produced in the oil B and V. In present study, lower CV of the pyrolytic oil may due to the incomplete conversion of sludge to fuel when lower pyrolysis temperature is applied.

In addition, compared to diesel oil and No. 6 fuel oil, oil A has CV of only 63 to 68 % of that for these fuels. The CV of the resulting oil however, is higher than the 23200 kJ/kg energy content in lignite and sub-bituminous coal [11] thereby reflecting the potential of this fraction as a liquid fuel. Besides being used as fuels, the oil may also be an important source of valuable chemical feedstock [5, 10].

Overall, the gas fraction represents the most important fraction. This is due to the pyrolysis conditions used in the experiments; high heating rates (about 118 °C/min) and elevated temperatures [3]. The results show that the gases yield increased proportionately with the heating time and temperature. At the beginning of the pyrolysis process, about 23% of the total yields were the gases. This maybe caused by the small amount of water and volatiles removed in the sludge at this stage. Significant increase in the gases yield was during the first 3 minutes of the treatment. At 650 °C, the gases yield was three fold of its initial production (about 67%, calculated by difference, dry basis). Longer treatment time will give rise to more production of gases [3].

CONCLUSIONS

Pyrolysis of sewage sludge can be conducted in a modified household microwave with input power of 700 W. Temperature achieved in the process was moderate, where temperature of 650°C was obtained after five minutes of microwave treatment. By means of microwave energy, drying and pyrolysis of wet sewage sludge was achieved in a single step. In order to carry out the pyrolysis of sewage sludge using microwave energy, the raw sludge must be mixed with a small amount of dielectric material such as graphite. This is because sewage sludge is a poor microwave receptor. Addition of graphite in the sewage sludge sample facilitates the increase in sludge temperature up to the necessary temperature for pyrolysis process to take place. Microwave pyrolysis of sewage sludge in a 700 W microwave oven at 650°C generated 27.33% char, 5.96% pyrolytic oil and 67% of gases. The pyrolytic oil has a calorific value of 28852 kJ/kg, higher than that of lignite and sub-bituminous coal (23200 kJ/kg) thereby reflecting the potential of this fraction as fuel material.

REFERENCES

[1] Ahmadun, F., and Alam, M.Z. (2002) Pretreatment of sewage treatment plant sludge by liquid state bioconversion for composting. In P.A. Gostomski and K.R. Morison (Eds.), Proceedings of the 9th APPChE Congress and CHEMECA (pp. 271.1-271.9). Christchurch: University of Canterbury.

[2] Inguanzo, M., Dominguez, A., Menendez, J.A., Blanco, C.G., and Pis, J.J. (2002) On the pyrolysis of sewage sludge: the influence of pyrolysis conditions on solid, liquid and gas fractions. Journal of Analytical and Applied Pyrolysis. **63**(1): 209-222.

[3] Menendez, J.A., Dominguez, A., Inguanzo, M., and Pis, J.J. (2004) Microwave pyrolysis of sewage sludge: analysis of the gas fraction. Journal of Analytical and Applied Pyrolysis. **71**(2): 657-667.

[4] Krietmeyer, S., and Gardner, R. (1996) Pyrolysis treatment. In J.R. Boulding (Ed.), EPA Environmental Sourcebook. (pp 375-382). Michigan: Ann Arbor Press, Inc.

[5] Dominguez, A., Menendez, J.A., Inguanzo, M., Bernad, P.L., Pis, J.J. (2003) Gas chromatographic-mass spectrometric study of the oil fractions produced by microwave-assisted pyrolysis of different sewage sludges. Journal of Chromatography A. **1012**(2): 193-206.

[6] Brigwater, A.V., Meier, D., and Radlein, D. (1999) An overview of fast pyrolysis of biomass. Organic Geochemistry. **30**: 1479-1493.

[7] Dominguez, A., Menendez, J.A., Inguanzo, M., and Pis, J.J. (2005) Investigations into the characteristics of oils produced from microwave pyrolysis of sewage sludge. Fuel Processing Technology **86**: 1007-1020.

[8] Committee on Microwave Processing of Materials. (1994) Microwave processing of materials. National Academy Press.

[9] Shinogi, Y., and Kanri, Y. (2003) Pyrolysis of plant, animas and human waste: physical and chemical characterization of the pyrolytic products. Bioresource Technology **90**(3): 241-247.

[10] Yaman, S. (2004) Pyrolysis of biomass to produce fuels and chemical feedstocks. Energy Conversion and Management. **45**: 651-671.

[11] Perry, R.H., Green, D.W., Maloney, J.O. (1999) Perry's chemical engineers' handbook. McGraw-Hill. New York.

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