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Studies reducing the H₂S from natural gas of using polyimide membrane

A laboratory pressure system with a membrane module was set up allowing to perform flow measurements and separation of gases and vapors. At the membrane polyimide module permeability test was conducted four mixes CH₄-H₂S concentrations ranging from 0.28% to 17.9% H₂S, with different ratios of the permeate flow to the retentate inlet pressures in the range of 10÷96 bar. It has been found that with increasing H₂S content in the inlet gas, methane losses decrease. Tests were carried out for different inlet gas flows at breakdown coefficients in the range of 0.08÷0.45. The study was conducted in a high pressure, aggressive environment for highly toxic gases. The article shows the possibility of changing the composition of product streams in the conducted membrane separation process. Three times the hydrogen sulfide content in the permeate was increased. The degree of H₂S removal from the inlet gas was up to 90%. It has been found that membrane techniques can be successfully used in the preliminary stage of the natural gas sweetening process.

Key words: membrane modules, gas separation, polyimides, hydrogen sulphide, natural gas.

Określenie możliwości separacji siarkowodoru ze strumienia gazów na membranach poliimidowych

Zestawiono laboratoryjną instalację ciśnieniową z modułem membranowym pozwalającą wykonywać pomiary przepływu i separacji gazów i par. Na module z membranami poliimidowymi przeprowadzono badania przepuszczalności czterech mieszanek CH₄-H₂S o stężeniach od 0,28 % do 17,9% H₂S, przy różnych stosunkach przepływu permeatu do retentatu dla ciśnień wlotowych w zakresie 10÷96 bar. Stwierdzono, że ze wzrostem zawartości H₂S w gazie wlotowym, maleją straty metanu. Testy prowadzone były dla różnych przepływów gazu wlotowego przy współczynnikach podziału na strumieniu w zakresie 0,08÷0,45. Badania prowadzono w wysokociśnieniowym, agresywnym środowisku dla gazów wysokotoksycznych. W artykule wykazano możliwość zmiany składu strumieni produktowych w prowadzonym procesie separacji membranowej. Uzyskano trzykrotny wzrost zawartości siarkowodoru w permeacie. Stopień usunięcia H₂S z gazu wlotowego dochodził do 90%. Stwierdzono, że techniki membranowe mogą z powodzeniem być zastosowane we wstępnym etapie procesu odsiarczania gazu ziemnego.

Słowa kluczowe: moduły membranowe, separacja gazów, poliimidy, siarkowodor, gaz ziemny.

Due to their structural, economic and environmental advantages, membrane techniques have the potential to replace or supplement some of the sorption methods used to date for the separation of gaseous components. INiG – PIB has been undertaking research in this field by conducting e.g. tests of the separation of natural gas components on a polyimide membrane [19, 33]. Subsequent tests have shown that it is possible to reduce the contents of N₂, CO₂ and H₂O in the gas and the high effectiveness of the removal of moisture from the gas on this membrane makes it possible to completely replace other more expensive drying methods [20]. This paper undertakes

to determine the possibility of separating hydrogen sulphide from gas on polyamide membranes.

H₂S content of the commercial gas is defined in Poland at 7 mg/Nm³ [37]. Achieving such a low level in one technological operation is possible only by means of chemical sorption methods, which are expensive in terms of investment and operation [16, 22, 29]. In this study, tests were carried out to isolate the hydrogen sulphide parts from natural gas by membrane separation method, which is characterised by very low operating costs.

Membrane separation of gaseous components is usually carried out by means of non-porous membranes [6, 23], where

the gas is adsorbed on the membrane surface and then diffuses across the barrier and desorbs on the other low-pressure side of the membrane as a permeate. The inlet gas stream loses the components that pass through the membrane faster after a contact with the membrane and is referred to as the retentate at the outlet. The separation of components is possible due to their permeability being different, which allows for obtaining two gas streams with different compositions. The permeability ratio of the two components is determined by selectivity (selectivity coefficient, separation coefficient).

The actual separation of the components is also influenced by the macroscopic factors related to the structure of the membrane modules, such as the contact time of gas at the high-pressure side with the membrane surface. The stream of gas flowing into the membrane module – F is separated from the stream passing through the membrane (permeate) – N . The ratio of the volume of the permeate (N) to the volume of the inlet gas is defined as the stage cut Θ :

$$\Theta = N/F \tag{1}$$

The selection of membrane consists in the application of such a membrane in which the separation of selected gas components will be as high as possible, with the permeate stream being of a satisfactory size. The lower stage cut Θ is, the more economical the expected effect of separation of the gaseous mixture into two streams is.

Membrane modules used for gas separation can have different designs. They can have a frame, plate, spiral-wound or tubular (including capillary) structures [4]. In industrial gaseous component separation processes, the most common

types of modules are those with spiral-wound and capillary hollow-fibre membranes [24, 32, 35].

The effectiveness and economy of the membrane separation of gaseous mixtures result from the properties of the used membrane (permeability and selectivity) and the gas flow rate [12, 17, 18]. If the application of membrane separation is aimed at lowering the mass content of H_2S in the gas, if it is a part of the technology for natural gas desulphurisation, i.e. the removal of e.g. 60÷90% H_2S from its stream, it has a serious economic justification [7, 31].

Acidic component separation methods using membranes have been in use for almost forty years. The commercialisation of most membrane tests has focused on the removal of carbon dioxide from natural gas and the mechanisms of H_2S permeation through membranes have been less investigated. The main reasons for this were the highly toxic and corrosive properties of this gas [14, 22, 26, 28]. Laboratory tests on the separation of H_2S from natural gas were started in published articles [6, 10, 21, 25, 31] by pilot tests on the separation of H_2S/CH_4 . Most of the results presented in the subsequent studies were related to membranes made of cellulose acetate (CA) [1–3], polyimide [11, 25, 27, 36], polyurethane [8, 23, 25] and Pebax [32]. Many studies concerned the modifications of polymer chains by promoting or inhibiting various function groups to achieve higher values of selectivity and permeability of the membranes made of these polymers [35]. Some publications refer to the modifications of polyimide membranes [9, 30], which are an attractive class of polymers and have a number of advantages (plasticisation resistance and swelling resistance) and high mechanical strength to high pressure differentials [11, 34, 36].

Test stand

The tests of the reduction of H_2S in natural gas were performed using a hollow-fibre capillary membrane module manufactured by UBE Industries (Japan). The membrane made of polyimide placed in the module is an example of an asymmetrical dense-porous membrane, which is shown in Figure 1.



Fig. 1. A section through the UBE polyimide membrane tested as part of laboratory tests [34]

These membranes are characterised by a unique structure resulting from the different length of the polymer chain with variable porosity in the cross-section. The module is placed in a high-pressure steel housing designed for counter-current stream flows, as shown in Figures 2 and 3.

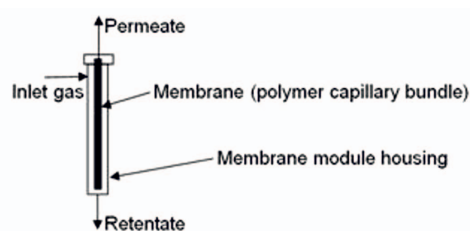


Fig. 2. The diagram of the membrane module in a counter-current system – one product stream (the permeate passing through the membrane) flows in the opposite direction to the other product stream (retentate)

The membrane module was the main part of the measuring system, which was equipped with flow and pressure control valves, thermometers and flow meters. The gas flowing into the module flowed into the space between the capillary membranes. The gas inlet was located on the permeate stream collection side (counter-current system).



Fig. 3. A photo of a hollow-fibre membrane bundle and a module housing for membrane separation

The gas separation tests of four different concentrations of hydrogen sulphide in methane were carried out on a membrane module supplied with gas from a cylinder. The mixtures of gases were prepared in INiG – PIB from pure components (>99.9%) by pumping them successively (first H₂S) in appropriate proportions into a hydrogen sulphide resistant to cylinder.

The system is equipped with gas regulators, ball (shut-off) valves, control valves and flow meters. A diagram of the system is shown in Figure 4.

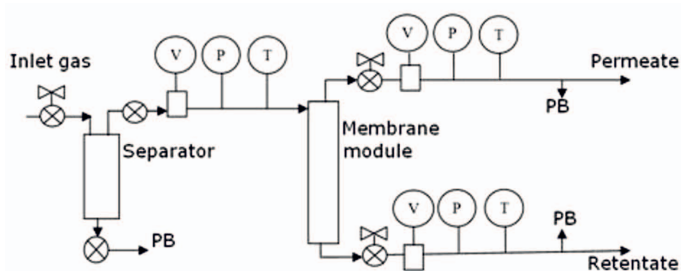


Fig. 4. The diagram of the system for testing the separation of methane – hydrogen sulphide mixture on a membrane module. Symbols: PB – sampling point, V – flow meters, P – pressure gauges, T – thermometers

The gas from the cylinder was directed to a small separator, which also performed the role of a pressure stabiliser (buffer), and then flowed through a needle valve into a flow meter whose readings were sent to a computer, where the current gas flow rate in l/min was read in appropriate software. The current gas pressure and temperature values were read before the inlet into

the membrane module. Pressure and temperature were measured again in the high-pressure product stream, and its flow rate was regulated by another regulator, behind which a pipette for collecting the sample for analysis was placed. The PVT measurements of the permeate stream were performed in a similar manner.

The configuration of the gas separation test stand containing a polyimide membrane module is shown in Figure 5. The regulation of the retentate outflow through the regulator and the needle valve made it possible to obtain different stage cut (Θ) values, which were calculated by simultaneous measurement of inlet gas, permeate and retentate flows.

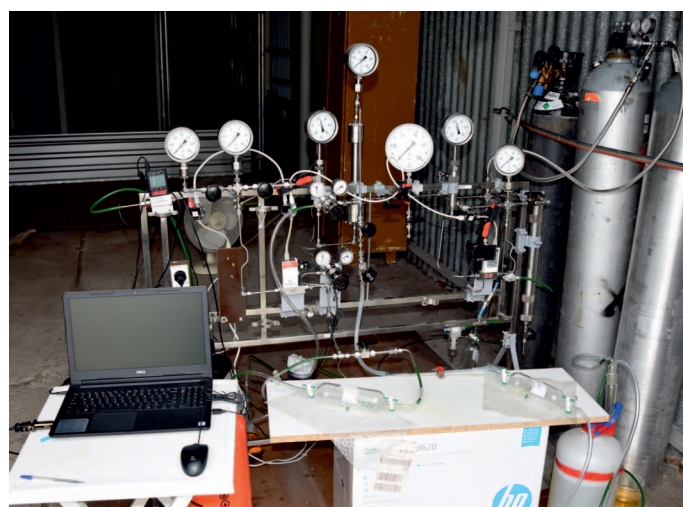


Fig. 5. Photos of the membrane gas separation test stand

Testing procedure

The H₂S–CH₄ separation tests started on a gas mixture with a very low hydrogen sulphide concentration of 0.28%. At such an H₂S concentration, the permeate flow rate depends mainly on the methane flow rate, which accounts for the majority of the gas in the mixture, as shown in Figure 6. In these tests, measurements were made at 20 bar and 35 bar. As shown in Figure 6, the permeate flow rate increases as the pressure increases. At a pressure of 20 bar, the changes in the permeate flow rate depending

on the inlet stream flow rate are not noticeable, while a slight increase in the permeate flow rate can be seen at a pressure of 35 bar. This is due to the visible presence of hydrogen sulphide (which penetrates through the membrane faster than methane).

The next two diagrams in Figures 7 and 8 show the changes of hydrogen sulphide concentrations in the product streams (P20, P35 – H₂S concentrations in the permeate, R20 and R35 – H₂S concentrations in the retentate).

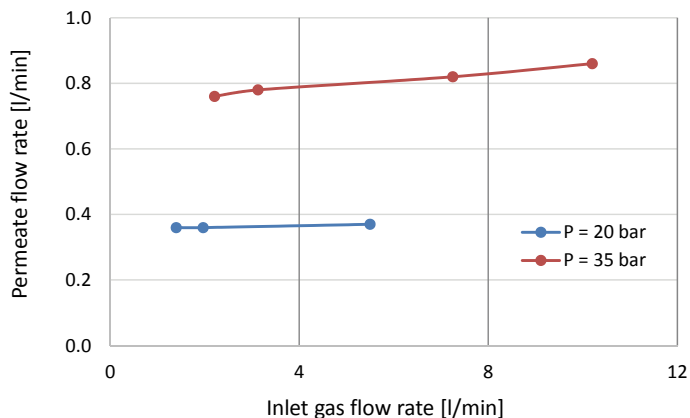


Fig. 6. The dependence of the permeate flow rate on the inlet gas flow rate for pressures: 20 and 35 bar

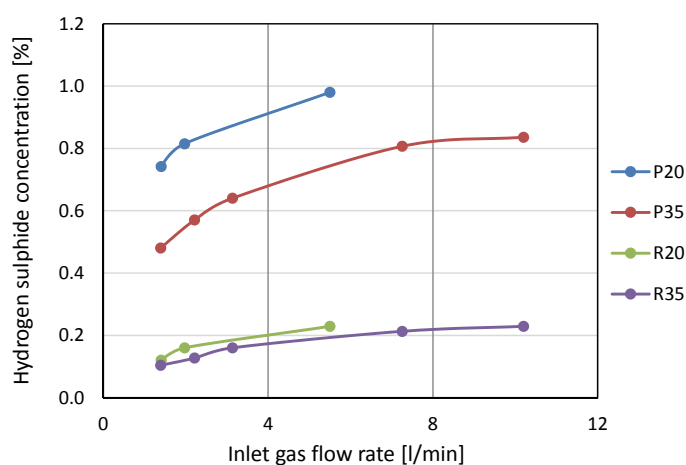


Fig. 7. The dependence of the hydrogen sulphide concentration in individual streams on the inlet gas flow rate for a pressure of 20 bar (P20 and R20) and 35 bar (P35 and R35)

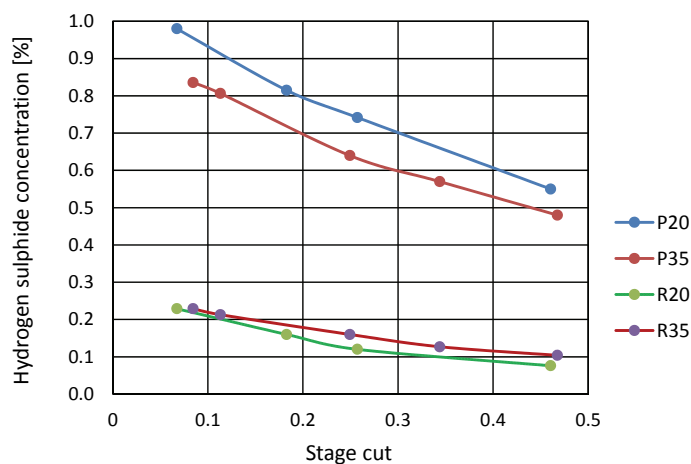


Fig. 8. The dependence of the hydrogen sulphide content in the permeate and retentate at 20 bar (P20 and R20) and 35 bar (P35 and R35) depending on the stage cut (the proportion of the permeate stream in the inlet gas)

The higher the inlet gas flow rate (and the lower the stage cut) is the higher the hydrogen sulphide content of both the streams is. For a pressure of 20 bar, the values of the hydro-

gen sulphide concentration in the permeate are higher than for 35 bar, while the reverse is true of the level of H₂S in the retentate. For low stage cut values, the hydrogen sulphide content in the permeate increased more than three times (from 0.28%). Based on the results above, the degree of hydrogen sulphide removal from the inlet gas was calculated, as shown in Figure 9.

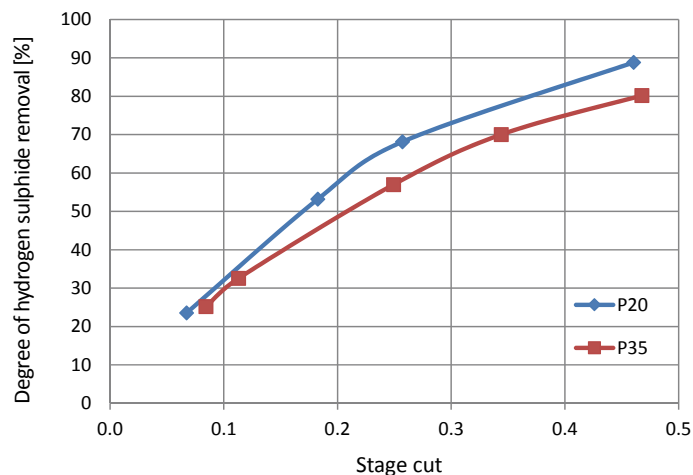


Fig. 9. The dependence of the degree of hydrogen sulphide removal from the inlet gas on the stage cut for the inlet gas pressure of 20 and 35 bar

As can be seen in Figure 9, with a longer gas contact time with the membrane (i.e. high stage cut values), the degree of removal of H₂S from the gas is high, but this happens at the expense of higher quantities of methane passing into the low-pressure permeate stream.

It should be remembered that while the process performance indicators may indicate their relative superiority at low pressures, the absolute amounts of hydrogen sulphide passing through the same membrane are higher at higher pressures. This is shown in Figure 10.

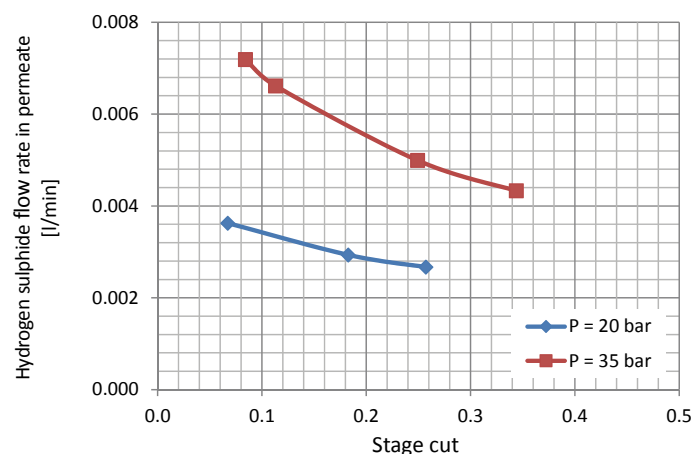


Fig. 10. The dependence of the quantity of the flowing hydrogen sulphide on the stage cut

The actual amount of H_2S flowing through the membrane is the product of its concentration in the permeate and the permeate flow rate.

Further tests were carried out for 2.9% of hydrogen sulphide in the inlet gas at separation pressures of 20, 60 and 80 bar. The results of the separation are shown in Figure 11.

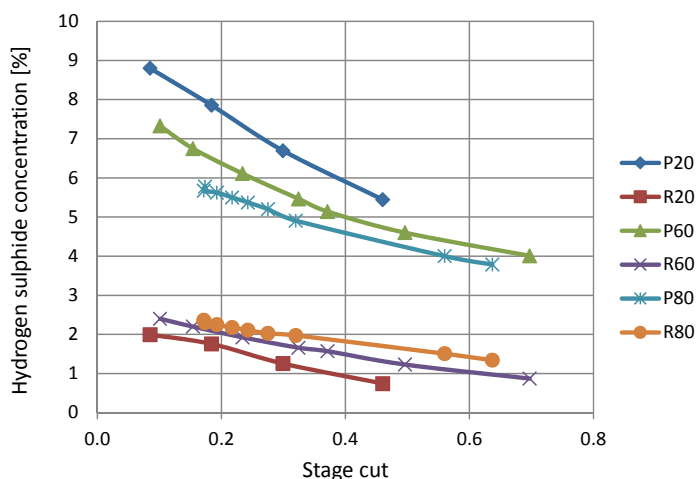


Fig. 11. The dependence of the hydrogen sulphide content in permeate and retentate at 20 bar (P20 and R20), 60 bar (P60 and R60) and 80 bar (P80 and R80)

As the tests show, the general trend of changes in concentrations of hydrogen sulphide in individual product streams is similar to that of the previous test series where the H_2S content was 10 times lower.

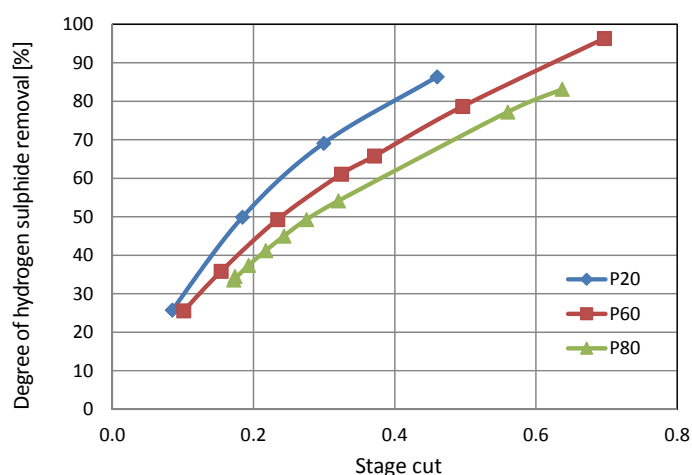


Fig. 12. The dependence of the degree of hydrogen sulphide removal from the inlet gas on the stage cut

The results of a series of separation tests on a polyimide membrane module for an inlet gas containing 7.5% of H_2S are shown in Figure 13.

The measurement results shown in Figure 13 were carried out for 10 bar, 40 bar and 80 bar. As can be seen from this diagram,

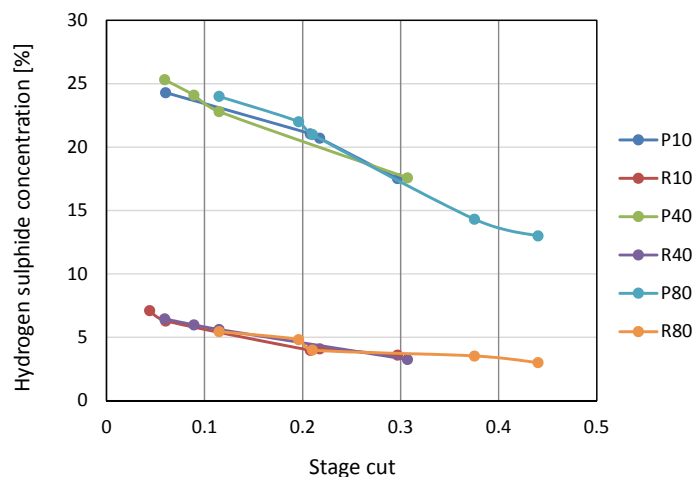


Fig. 13. The dependence of the hydrogen sulphide content in the permeate and retentate for 10 bar (P10 and R10), 40 bar (P40 and R40) and 80 bar (P80 and R80) – 7.5% H_2S inlet gas

the differences between the measurements at different pressures are minimal, which sets them apart from the previous tests on gas mixtures with lower hydrogen sulphide content in the gas.

The last series of tests was performed for an inlet gas stream containing 17.9% of hydrogen sulphide and 82.1% of methane at different separation pressures. The measurement results for this gas is shown in Figure 14.

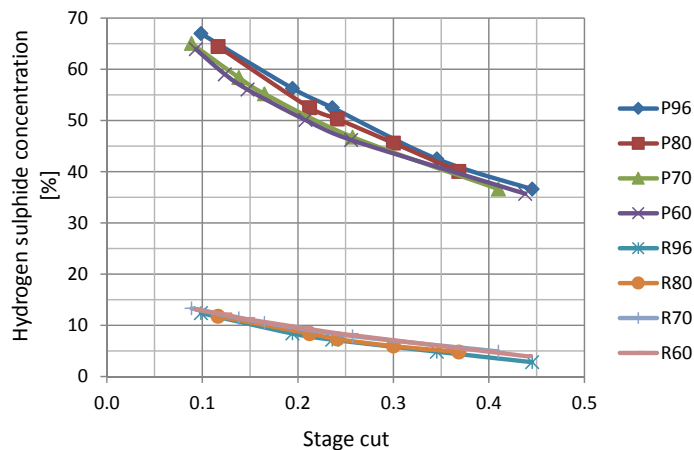


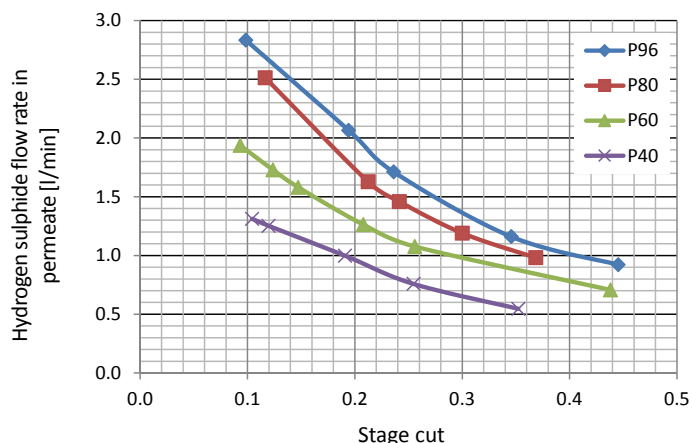
Fig. 14. The dependence of the hydrogen sulphide content in the permeate and retentate on the stage cut for the inlet gas pressure of 60 bar (P60 and R60), 70 bar (P70 and R70), 80 bar (P80 and R80) and 96 bar (P96 and R96). Inlet gas – 17.9%.

Like for the inlet gas containing 7.5% of H_2S , in the case of the 17.9% hydrogen sulphide gas shown in Figure 14, the differences in its content in the product streams at different pressures are small. However, it can be seen in the permeate that the increase in hydrogen sulphide concentration in the inlet gas caused a change in the trend in its content together with the pressure increase. For low stage cut values, the hydrogen sulphide content of the permeate exceeds 60%.

Changes in the hydrogen sulphide flow rate in the permeate depending on the stage cut are shown in Figure 15.

Figure 15 shows that the dependence of the hydrogen sulphide content on the flow rate depends less on the process pressure than in the previous tests with lower concentrations of hydrogen sulphide.

Fig. 15. The dependence of the quantity of flowing hydrogen sulphide in the permeate on the stage cut at pressures of 96, 89, 60 and 40 bar



Conclusions

The tests were conducted for four CH₄-H₂S gas mixtures with 0.28%, 2.9%, 7.5% and 17.9% hydrogen sulphide concentrations. Tests were carried out at various inlet gas pressures ranging from 10 bar to 96 bar. In the main part of the tests, permeate pressures were collected at a low excess pressure (0.06 to 0.1 bar), which was associated with the resistance to flow through the washers absorbing hydrogen sulphide. All the tests were carried out at 21±22°C.

The tests were carried out for different inlet gas flow rates with the stages cut values mostly ranging from 0.08 to 0.45, which means that 8÷45% of the inlet gas stream passed through the membrane.

In all the tests, the permeate flow rate increases as the pressure rises. Similarly, the permeate flow rate increases with an increase in the flow rate of the high-pressure stream, with the rate of this increase being higher for higher hydrogen sulphide concentrations. The permeate flow rate decreases with the increase in the stage cut, which is due to the decrease in the content of the faster-permeating component (H₂S) along the

capillaries in the counter-current membrane module. According to the analysis of the tests, as the H₂S content in the inlet gas increases, methane losses decrease, e.g. for the stage cut of 0.2, regardless of the pressure conditions of the conducted tests (from 20% for 0.28% H₂S concentration to approx. 12÷13% for 17.9% of H₂S).

In the case of the desulphurisation of an actual natural gas, the mixture will also contain other components that pass through the membrane together with methane. This is especially true for nitrogen and carbon dioxide, which permeate through the polyimide membrane much faster than methane. This means that in the process of desulphurisation of actual natural gas on membranes, the methane losses will be proportionally lower because it will be N₂ and CO₂ (rather than methane) that will pass into the permeate to a large extent.

To sum up, membrane techniques can be successfully applied in the natural gas desulphurisation process at the initial stage of mass removal of hydrogen sulphide prior to the process of thorough purification by other methods.

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