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The Use of Equipment for the Study of Phase Changes to Determine the Conditions of Precipitation of Inorganic Sediments in Geothermal Waters

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Abstract: In Poland, there are low-temperature geothermal reservoirs that can be used for various purposes in many regions of the country. Low-temperature deposits of geothermal waters are common and occur much more frequently than high-temperature deposits. They contain water with temperatures lower than 150 °C. Their temperature normally ranges from 20 to 90 °C. Achieving a state of equilibrium depends on many factors, including the kinetics of reactions between the individual components of the system, temperature, reactivity of reservoir rock, concentration of chemical components in the water and the time the water remains in contact with the rock. Therefore, this article presents the possibility of checking the conditions of precipitation of inorganic sediments in geothermal waters with the use of PVT equipment. Tests were carried out with the use of geothermal waters under given dynamic conditions (pressure, temperature and flow). This paper confirms the suitability of using the equipment for PVT (device for the study of phase changes) testing in order to determine the conditions for the precipitation of inorganic sediments in geothermal projects. Tests on the precipitation of solid sediments in geothermal waters were carried out. A result of the research is the adaptation of the equipment for PVT testing in order to determine the conditions for the precipitation of inorganic deposits in geothermal waters. As a result, different capillary blocking times were obtained depending on the measurement conditions (e.g., for P = 40 bar, T = 120 °C and q = 1 cm^3/min, the blocking start time was 10.8 min). The authors found that solid sludge inhibitors, as well as other chemicals used, including paraffin inhibitors, must be periodically adjusted to prevent precipitation in geothermal waters.

Keywords: deposits; geothermal; water

1. Introduction

This work aimed to determine the possibility of using the research infrastructure owned by the Institute to solve problems related to ensuring an appropriate flow rate during the extraction and reinjection of geothermal water. In particular, the focus was on the precipitation of inorganic deposits [1].

Geothermal energy is classified as a renewable energy source (RES). This is because of its particularly important features: independence from changing climatic and weather conditions, relatively easy control and the possibility of using it without causing major disturbances to the natural environment. As more and more decisive measures must be taken to reduce greenhouse gas emissions, the position of geothermal energy in the energy transformation seems to be getting stronger. Many experts see geothermal energy as an essential part of the world's "green deal" [1].

Geothermal systems exist all over the world in various geological conditions, but so far, they have been of practical importance only in regions with a high geothermal gradient, mainly related to the boundaries of tectonic plates.

Currently, thanks to significant progress in technology and the need to increase the share of renewable energy sources in the energy mix, drilling in areas with normal and low geothermal gradient is gaining momentum [1,2].

So far, no internationally uniform standards for the classification and reporting of geothermal resources have been developed. The discrepancies concern both the nomenclature/definitions and the rules for evaluating geothermal resources [3]. Geothermal systems can be classified based on the reservoir temperature, enthalpy, condition phase of geothermal media, their nature and geological conditions. The total amount of thermal energy stored in the earth's crust at a given distance in relation to a specific area and the annual average surface temperature is called a geothermal resource [4].

The most common criterion for classifying geothermal resources is the temperature (enthalpy) of the media carrying heat. Due to the nature of the geothermal reservoir and the heat-transfer medium, two types of geothermal energy resources can be distinguished:

- Hydrothermal energy, where the energy is stored in groundwater, and is extracted in the form of water, steam or water-steam mixtures;
- Petrothermal, where the energy is stored in dry, heated porous rocks or salt domes, and is acquired by injection of media (gas or water) from the surface to the geothermal reservoir.

The research carried out so far has been carried out with the use of equipment specially dedicated to this purpose. In general, artificially produced brines were also used. So far, the apparatus for PVT testing has not been adapted to determine the conditions for the precipitation of inorganic sediments in geothermal waters.

1.1. Geothermal Energy in Poland

Poland is situated outside the areas of modern tectonic and volcanic activity; therefore, the occurrence of geothermal energy is related to waters with temperatures usually not exceeding 90 °C, and in rare cases slightly exceeding 100 °C, which can be classified as low-temperature resources (resources with a low enthalpy) [5]. According to Polish regulations, thermal (geothermal) waters are groundwaters used as a heat carrier, which at the outlet have a temperature of not less than 20 °C [6].

Geothermal resources in Poland are mainly related to the thermal waters in the Mesozoic formations in the Polish Lowlands, the Inner Carpathians (Podhale), the Carpathians and the Subcarpathian Depression. The underground waters of the Palaeozoic formations located in the Outer Carpathians and Sudetes are of minor importance [7,8]. One of the methods of earth heat extraction that is used in Poland to an increasing extent is geothermal heat pumps. It is estimated that the number of geothermal heat pumps installed in our country is approximately 70,000, and their total capacity is 650 MW (as of 2019). In 2020, Poland was ranked eighth in Europe with respect to the number of geothermal heat pumps installed [9].

In recent years, there has been an increase in the use of land heat in Poland. This is related to the implementation of a strategic goal for the Polish economy—increasing the share of renewable energy sources in the energy balance of Poland. In order to stimulate the development of national geothermal energy, the National Fund for Environmental Protection and Water Management has so far implemented three priority programs:

- "Geology and mining. (Part 1) Getting to know the geological structure of the country and the management of mineral and groundwater resources".
- "Polish Geotermia Plus".
- "Providing access to thermal waters in Poland" [10].

As part of the latter, the National Fund for Environmental Protection and Water Management will finance the drilling of geothermal holes in 15 localities throughout the country.

In 2015–2019, the use of geothermal energy was gradually increasing: in 2019 its consumption was 15.6% higher than the consumption in 2015. Geothermal energy was used to meet the demand for heat in 2019—75.0% of consumption in households and 25.0% in trade and services [11].

It is expected that the increase in the use of geothermal energy in Poland will be more and more dynamic, especially in terms of installations of higher heating capacity (geothermal heating plants). Energy extraction from natural geothermal systems is not a completely hassle-free process. One of the major challenges when it comes to geothermal water extraction, in addition to corrosion, is the problem of the precipitation of inorganic sediments in geothermal installations, especially in points where there are changes in the physical and chemical parameters of water, e.g., heat exchangers and areas near the reinjection boreholes, etc. All these questions are discussed in the next subsection.

1.2. Problems Related to the Use of Geothermal Waters

All geothermal waters contain dissolved solids. The amount varies significantly: from about 100 g/ton to even 250,000 g/ton. Dissolved substances remain in a state of thermodynamic equilibrium under given (reservoir) conditions of pressure and temperature, which also depends on the lithology of rock, the amount of components outwashed by the water from the surrounding rocks and entering the solution, the water circulation conditions and the process of secondary mineral formation. Achieving a state of equilibrium depends on many factors, including the kinetics of reactions between the individual components of the system, temperature, reactivity of the reservoir rock, concentrations of the chemical components in the extracted water and the time the water remains in contact with the rock [12].

During extraction, due to changes in pressure and temperature, the steady-state equilibrium breaks down, which may lead to the precipitation of the substances dissolved in the water. Most substances have a lower solubility at lower temperatures. The breakdown of the equilibrium may also occur as a result of changes in the water chemical mechanism during the flow of fluid to the extraction borehole, or reinjection through the reinjection borehole. Additionally, geothermal fluids may contain components that are aggressive to steel [13].

Therefore, obtaining thermal energy in geothermal heating plants may be significantly difficult due to the occurrence of unfavorable phenomena, both in geothermal doublet boreholes and in borehole zones, as well as in the surface systems. These include the corrosion of pipes and devices, a decrease in efficiency caused by clogging of the equipment near the boreholes, and, above all, the accumulation of sediments precipitated from geothermal water on the downhole casing and surface equipment. These problems are common in almost all geothermal heating plants. They can have serious economic consequences, resulting from energy losses, increased capital expenses due to oversizing of the system, increased injection costs (increased pump capacity), cleaning and reconstruction costs, decreased operating efficiency or even liquidation of extraction or injection boreholes due to clogging [14,15].

The composition of sediments accumulating in geothermal installations is usually quite complex and results from both the chemistry of groundwater and the influence of factors such as: deposit conditions (pressure and temperature), history of water–rock interaction and extraction parameters.

Calcium carbonate usually precipitates from low- and medium-temperature thermal waters ($T < 150$ °C). In cases where the waters contain a relatively high amount of iron and dissolved sulfides, additionally there may be deposits of iron sulfides. Waters with high enthalpy and low salinity are characterized by the presence of silica deposits, while in high-temperature waters with a high content of dissolved components, both silica sediments and sulfide sediments, represented mainly by lead sulfide, appear [16–20].

The high complexity of sediment formation is due to the large number of substances dissolved in geothermal waters and the variety of physical mechanisms that may be involved, such as a transfer of mass, heat and momentum or chemical reactions on the surface of the plant equipment. Moreover, the high variability in the composition of geothermal waters at individual locations and during the inflow of waters from the reservoir to the surface installations make it difficult to formulate universal statements on the causes (mechanisms) and preventive measures.

2. Materials and Methods

2.1. Tube Blocking Tests (TBT)

The Tube Blocking Test consists in simultaneously pumping two types of brine with an appropriate flow rate through a capillary with specific parameters (length and internal diameter), under given pressure and temperature conditions (PT). The heating of the brines flowing in separate pipes is executed in a thermostat. There is also a capillary into which both brines are pushed after mixing. The differential pressure (ΔP) over the entire length of the capillary, i.e., from the mixing point at the inlet to the outlet of the pipe, is monitored. Precipitation causes an increase in ΔP in the capillary, which is recorded on a plot as a function of time [21].

The test is performed according to the procedure established by NACE (NACE International Publication 31105. Dynamic Scale Inhibitor Evaluation Apparatus and Procedures in Oil and Gas Production). The procedure defines all the parameters of the testing equipment and the conditions under which the measurements are to be performed.

The internal diameter of the capillary should be within the scope of 0.05 mm to 1.7 mm. The capillary should be made of 316 SS. The length of the capillary should be between 50 mm and 15 m [22–25].

The testing temperature is generally between 70 °C and 90 °C. However, if the temperature is at the point where sediment problems occur, it should be adapted to the actual conditions.

The pressure level required during the test is not clearly defined. The choice of pressure depends on the capabilities of the equipment and the assumptions made by the worker [26,27].

The basic materials used for this research were geothermal water samples. These brines were drawn directly from the boreholes on the site. The chemical composition of the brines is presented in Table 1.

Table 1. Chemical composition of geothermal waters used for research.

Parameters	Unit	Water A	Water B
Reaction	pH	7.5	7.1
Chlorides	mg/L	483	478
Bromides	mg/L	0.88	0.88
Sulfates	mg/L	1060	950
Sodium	mg/L	520	490
Potassium	mg/L	51	49
Magnesium	mg/L	47	45
Calcium	mg/L	230	230
General mineralization	mg/L	2650	2550

Since, after mixing the waters no solid sediment precipitation was found, one of the water types was modified. Sodium bicarbonate was added to water "A". As a result, after mixing the brines under the given pressure and temperature conditions, the precipitation of solid deposits was observed, and thus the capillary was blocked.

2.2. Description of the Apparatus Used for the Research and the Research Methodology

The research on the precipitation of inorganic sediments in geothermal waters was carried out with the use of a mercury-free PVT apparatus for testing the phase properties of reservoir fluids. The basic element of the mercury-free PVT apparatus is two pressure chambers. Before starting the water test, about 400 cm^3 of water "A" was introduced into this chamber.

Temperature and pressure were measured simultaneously via an integrated transducer (P-T) designed in such a way that both sensors (pressure and temperature) are integrated into a highly resistant compact housing. The transmitters are factory tested and calibrated, and the results of these calibrations are entered in the form of appropriate tables into the computer software supporting the apparatus [28].

The above-presented apparatus was used to determine the conditions for the precipitation of inorganic sediments in geothermal waters. To perform these tests, the PVT apparatus was appropriately expanded with several high-pressure connections and an ultrathermostat to maintain the required temperature (Figure 1).

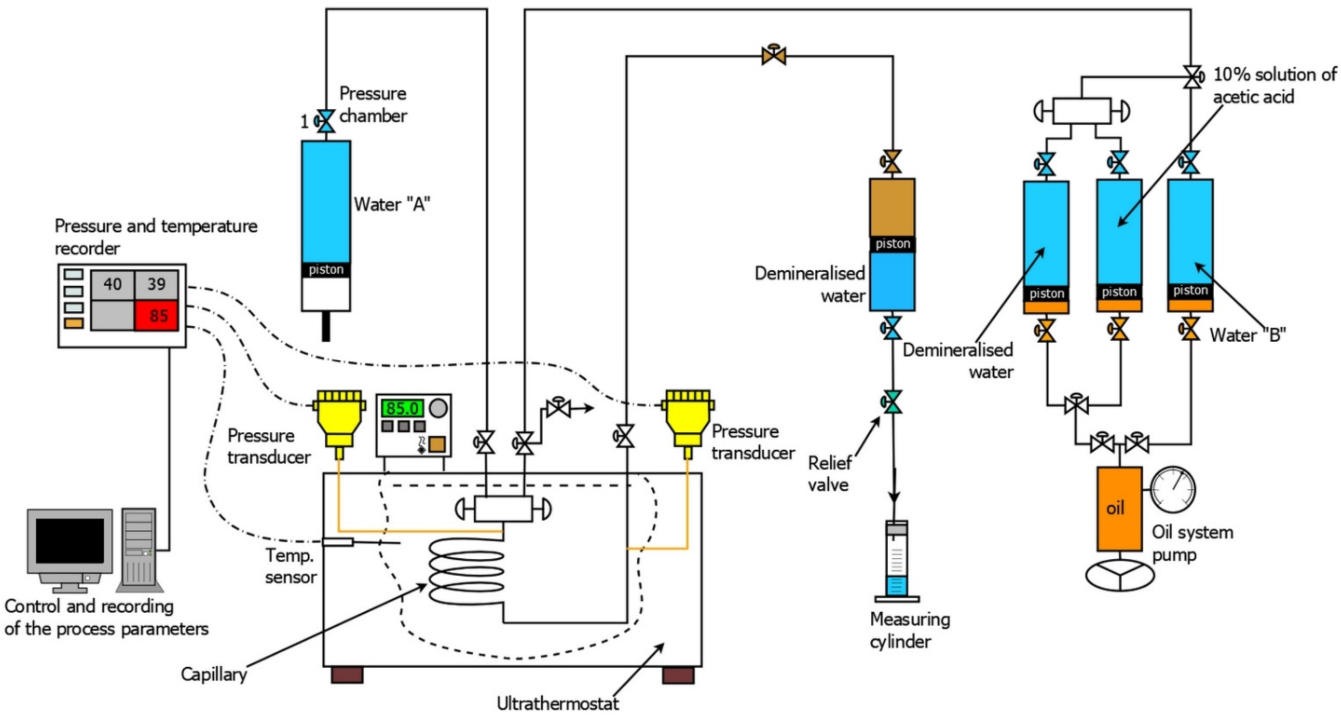

Figure 1. Measuring system diagram.

The main element of the apparatus required to determine the conditions for the precipitation of inorganic sediments from geothermal waters is a capillary made of 316 SS steel. According to the NACE procedure, the capillary should be 2.3 m long and have an internal diameter of 0.9 mm. The upgrade of the apparatus consisted in installing this capillary in an ultrathermostat heating oil bath. Use of the ultrathermostat additionally enabled precise adjustment of the flowing brine's temperature (water types marked as "A" and "B"). Before starting the research, geothermal waters were filtered to exclude any solid contaminants that could have an impact on the measurement. As mentioned earlier in the study, water "A" was introduced into the PC chamber of the test apparatus, while water "B" was poured into the pressure container. Waters "A" and "B" came from two different boreholes.

Pressure transducers were installed upstream and downstream of the capillary to determine the pressure difference. By using such a solution, it was possible to constantly record the pressure and its variation during the test. The measurements were saved directly on a memory card so that the values could be read out later. To facilitate the research work, several high-pressure connections had to be established. One of the main elements was the supply and connection of both brines. The problem of air pockets in the pipes had to be eliminated, and an additional vent valve was used for this purpose. After the samples were fed to the mixing valve, the valves were opened and the two waters were connected. After starting pumping of the brines under certain pressure and temperature conditions with a given flow rate, the pressure in the system increased. The pressure was lowered by opening the relief valve.

After passing through the capillary, the mixed brine solution was discharged under pressure through the return valve into the discharge container.

Once the measurement was complete, the entire system was washed. For this purpose, 10% acetic acid solution was used, which was forced through the capillary to thoroughly remove the inorganic deposit. Upon completion of this operation, the system was washed again with distilled water to remove acetic acid. Once the entire system was washed, the next measurement was started by repeating the entire test procedure.

The diagram of the procedure is presented in Figure 2.

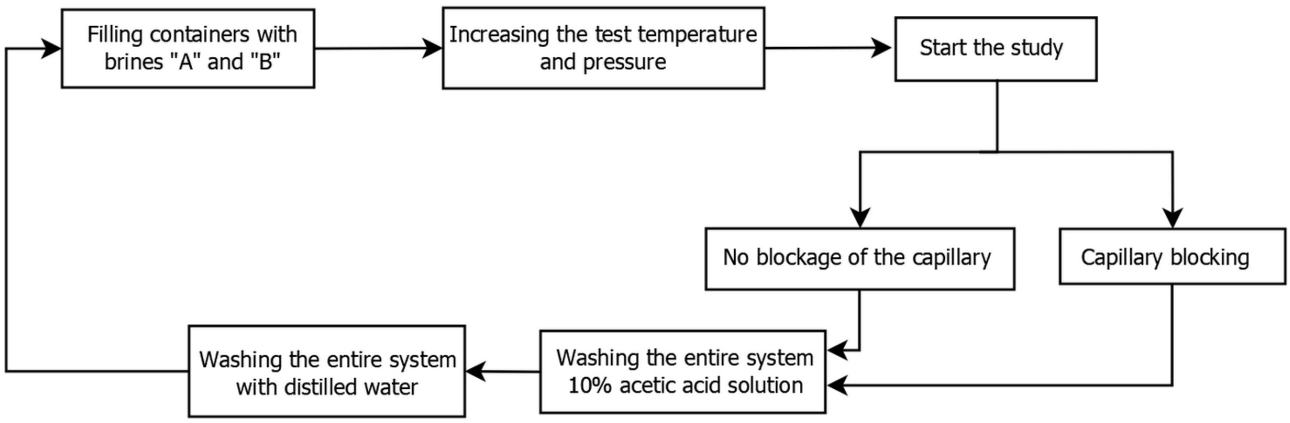

Figure 2. Research flowchart.

3. Results

3.1. Research with the Use of Geothermal Waters

As mentioned earlier, the geothermal waters drawn from two boreholes located on site were used (brine "A" and "B").

The possibility of precipitation of inorganic sediments at the actual deposit temperature was checked in the first place. The temperature was averaged and set at the level of 85 °C, whereas the pressure was set to 40 bar. All parameters were determined in accordance with the procedure provided by NACE. Based on previous experience (research on calcium carbonate inhibitors [29] and barium and strontium inhibitors [21]), as well as the capabilities of the research equipment possessed by the authors (limited flow rate of brines), the maximum flow rate was assumed to be at the level of 2 cm^3/min for each of the water types (Figure 3). The tests were carried out at a pressure of 40 bar and temperatures of 85 °C and 120 °C.

Despite pumping the brines through the capillary for a long time (from 130 to over 160 min), no significant increase in pressure in the system was observed. At a temperature of 85 °C, i.e., the medium reservoir temperature, the pressure increase did not exceed 0.5 bar; therefore, it was decided to increase the temperature significantly. The system pressure remained the same. It has no significant effect on the precipitation of solid deposits [24]. Despite increasing the temperature to the level of 120 °C, there was no precipitation of inorganic deposits in the capillary. Only the pressure slightly increased by 0.5 bar. Such a slight increase may be caused by the flow resistance, as well as the measurement inaccuracy of the pressure-measuring transducers. In order to investigate the precipitation of inorganic sediments in geothermal waters, it was decided to modify one of the water types by adding sodium bicarbonate.

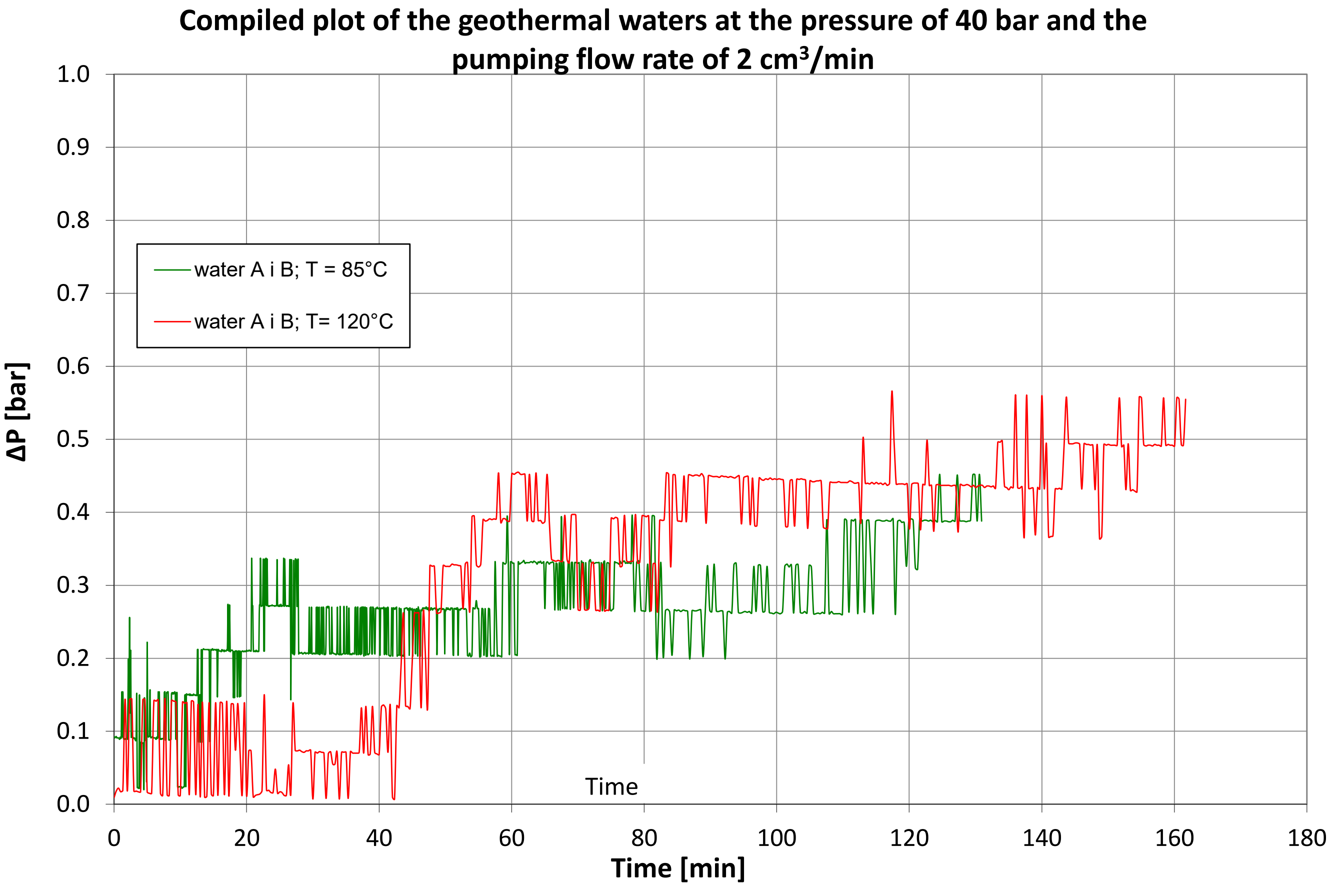

Figure 3. The course of the study of individual geothermal water types (measurement error is ±4%).

3.2. Tests on Geothermal Water (Modified with 2 g of Sodium Bicarbonate per 800 mL)

To 800 mL of the geothermal water "A", 20 g of sodium bicarbonate was added, and then the solution was stirred for 24 h (Figure 4). The mixture prepared in this way was filtered and then poured into the testing chamber. Water "B" was introduced into the second chamber. The series of measurements were carried out at two temperatures (85 °C and 120 °C). At a given temperature, the measurements were taken at pressures of 18 and 40 bar and flow rates of 1 and 2 cm^3/min for each water type. For a temperature of 85 °C, the time until the capillary became blocked was between 23 and 48 min, while in the case of 120 °C, this time decreased to about 9–20 min. This shows the impact of temperature on the blocking time of the capillary. The graphs also show the different nature of the curves depending on the pressure level. At 40 bar, the pressure increased more rapidly, which means that deposits are "built up" in the tube faster, while at a pressure of 18 bar, the increase is much slower. This is because the accumulation of inorganic deposits on the capillary walls is much slower.

3.3. Tests on Geothermal Water (Modified with 1 g of Sodium Bicarbonate per 800 mL)

After conducting the series of tests described above, the authors decided to lower the concentration of sodium bicarbonate in water "A" to the level of 1 g/800 mL. As in the previous case, the solution was stirred for 24 h and then filtered. Initially, the mixture was tested at a temperature of 85 °C, a pressure of 40 bar and a flow rate of 1 cm^3/min for each of the water types. Capillary blocking occurred after almost 150 min. In this case, the authors decided to increase the temperature to 120 °C and perform another series of tests. The tests were carried out at pressures of 18 and 40 bar and flow rates of 1 and 2 cm^3/min for each of the water types. At 40 bar, blocking occurred after approximately 31 min (for the flow rate of 1 cm^3/min) and after 26 min (for the flow rate of 2 cm^3/min), whereas in the case of a pressure of 18 bar, blocking occurred after 42 and 36.5 min, respectively. As can be seen in Figure 5, for a pressure of 40 bar, the increase in delta P occurred much faster and more rapidly than for a pressure of 18 bar. This is due to the higher test pressure. The course of the individual tests is shown in Figures 6 and 7.

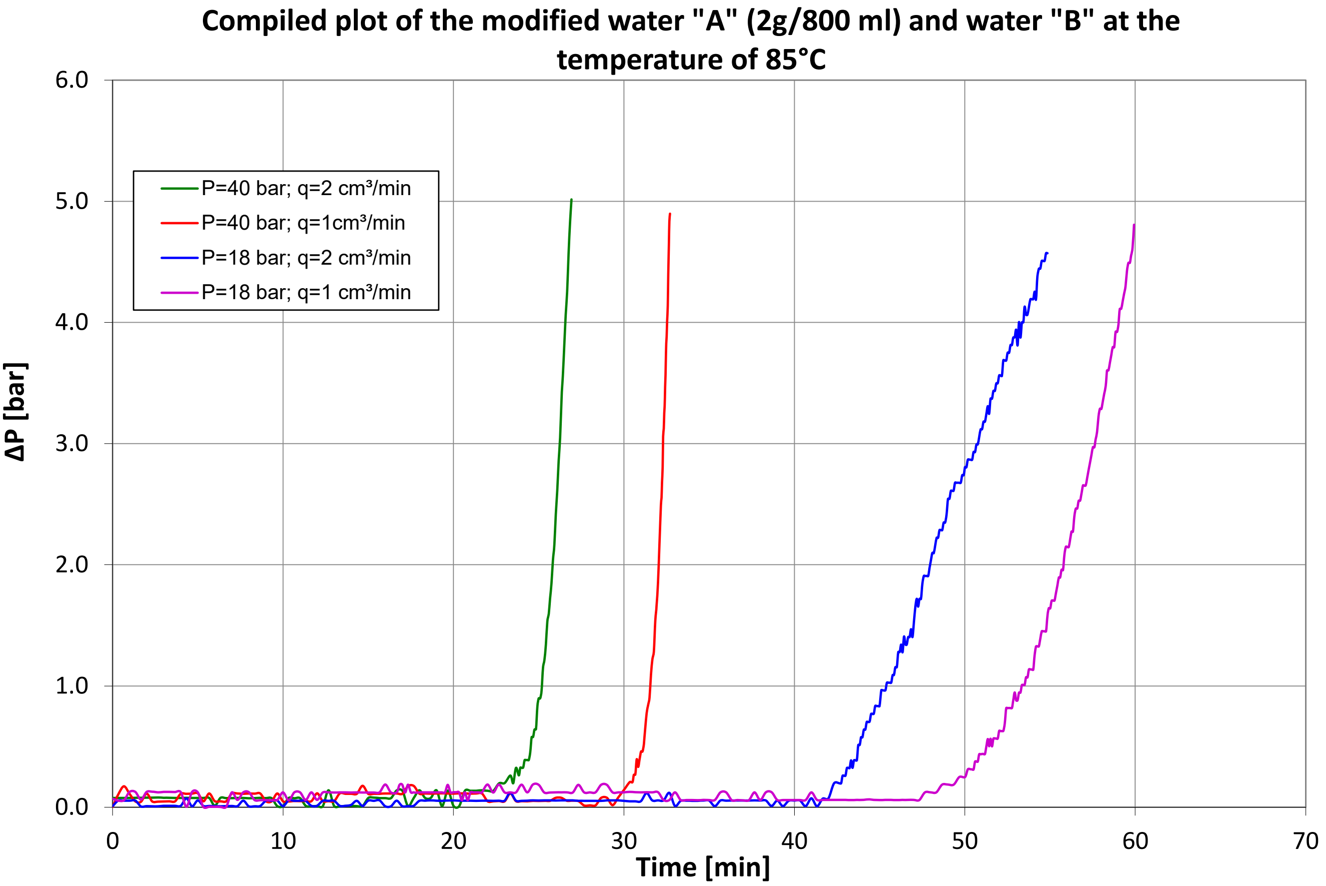

Figure 4. Tests for the precipitation of sediments for water "A" modified with sodium bicarbonate (2 g/800 mL) and water "B" at a temperature of 85 °C (measurement error is ±4%).

3.4. Long-Term Test for Geothermal Waters "A" and "B"

After performing all the above-mentioned test series, the authors decided to perform a long-term test. It resulted from the fact that in previous tests the pumping pressure was exceeded by 0.5 bar and there was a possibility that the capillary could become blocked after longer pumping in such conditions. As the chambers with both types of water did not have such a large capacity, the pumping was stopped during the test and the waters were entered again into the containers. Throughout the entire duration of the test and refilling of the brines, the measurement conditions were not disturbed (pressure at the level of 40 bar, temperature at the level of 120 °C). The flow was maintained for about 6.0 h. During this time almost 1400 cm^3 of geothermal water was pumped in total through the testing circuit. Unfortunately, as in the previous series of tests, no significant increase in the pressure difference was found. Delta P was slightly above 0.4 bar. After a series of tests, it can be stated with certainty that the geothermal waters drawn for the present research did not show the inorganic sediment precipitation tendency (Table 2). The characteristics of the performed test are shown in Figure 8.

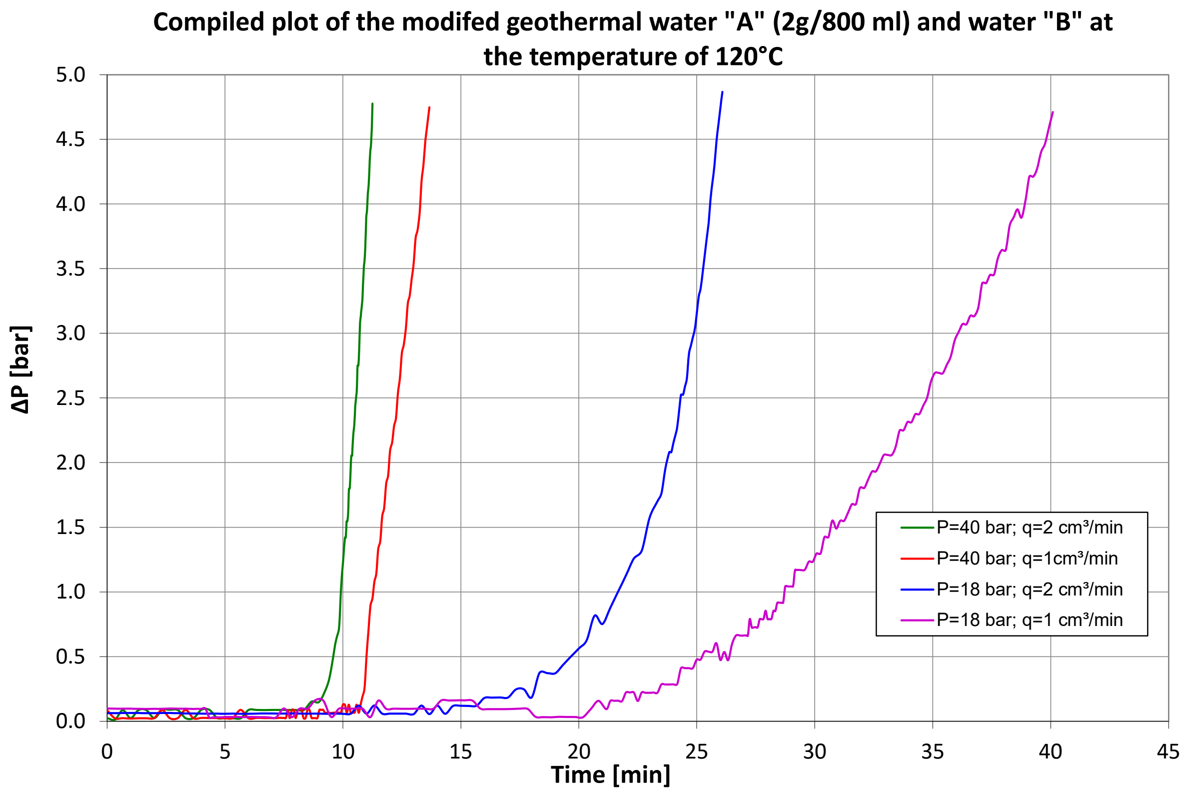

Figure 5. Tests for the precipitation of sediments for water "A" modified with sodium bicarbonate (2 g/800 mL) and water "B" at a temperature of 120 °C (measurement error is ±4%).

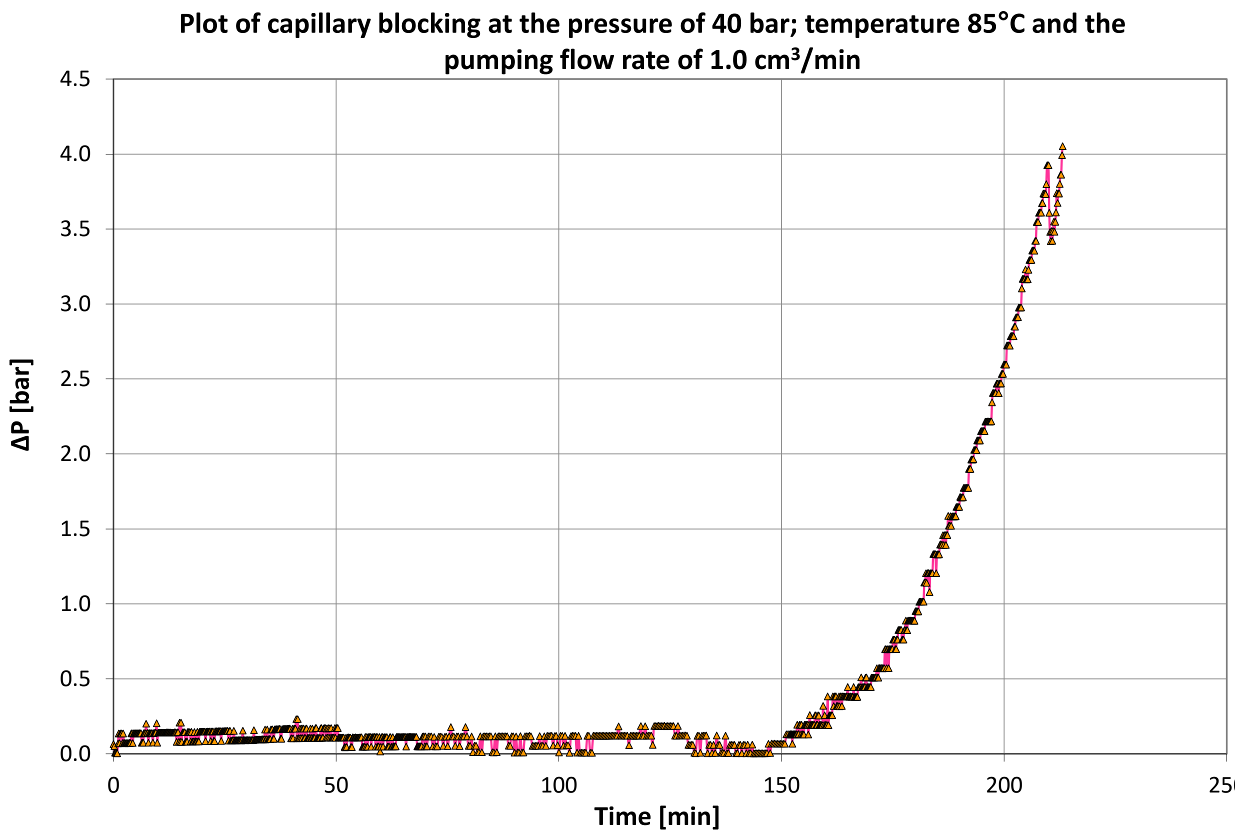

Figure 6. Tests for the precipitation of sediments for water "A" modified with sodium bicarbonate (1 g/800 mL) and water "B" at a temperature of 85 °C (measurement error is ±4%).

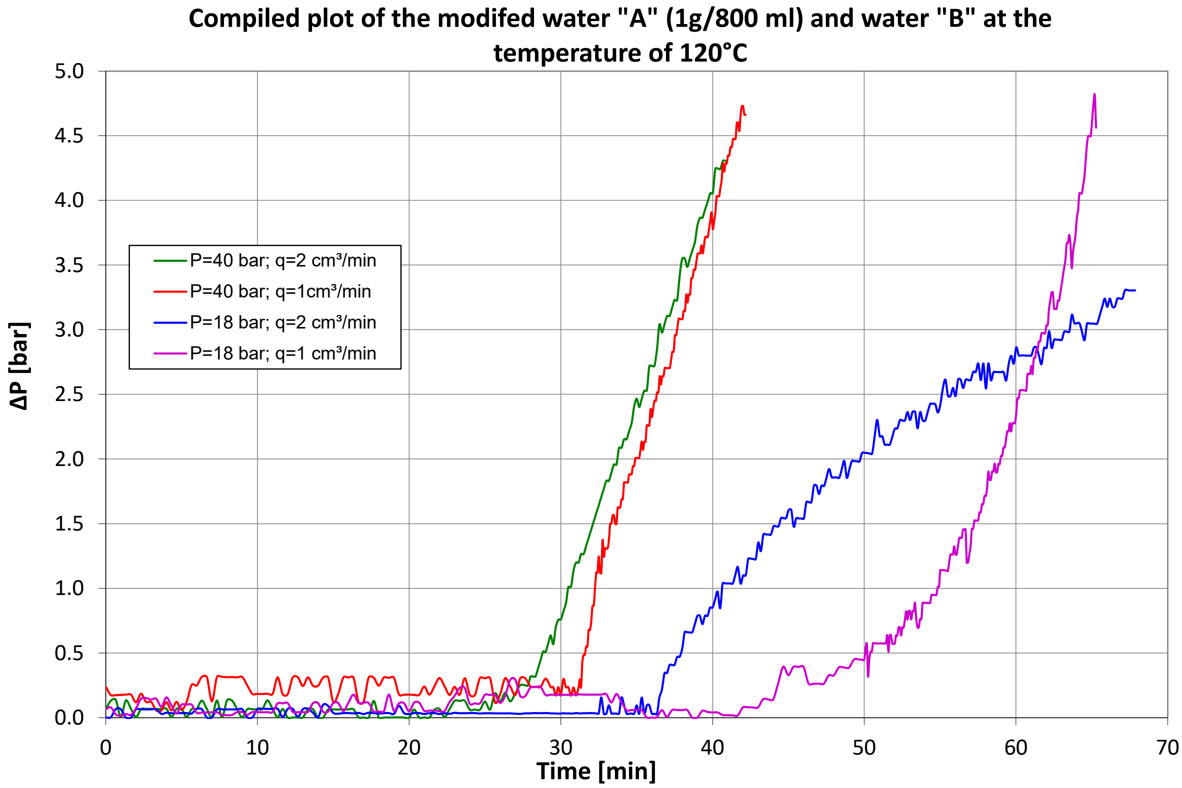

Figure 7. Tests for the precipitation of sediments for water "A" modified with sodium bicarbonate (1 g/800 mL) and water "B" at a temperature of 120 °C (measurement error is ±4%).

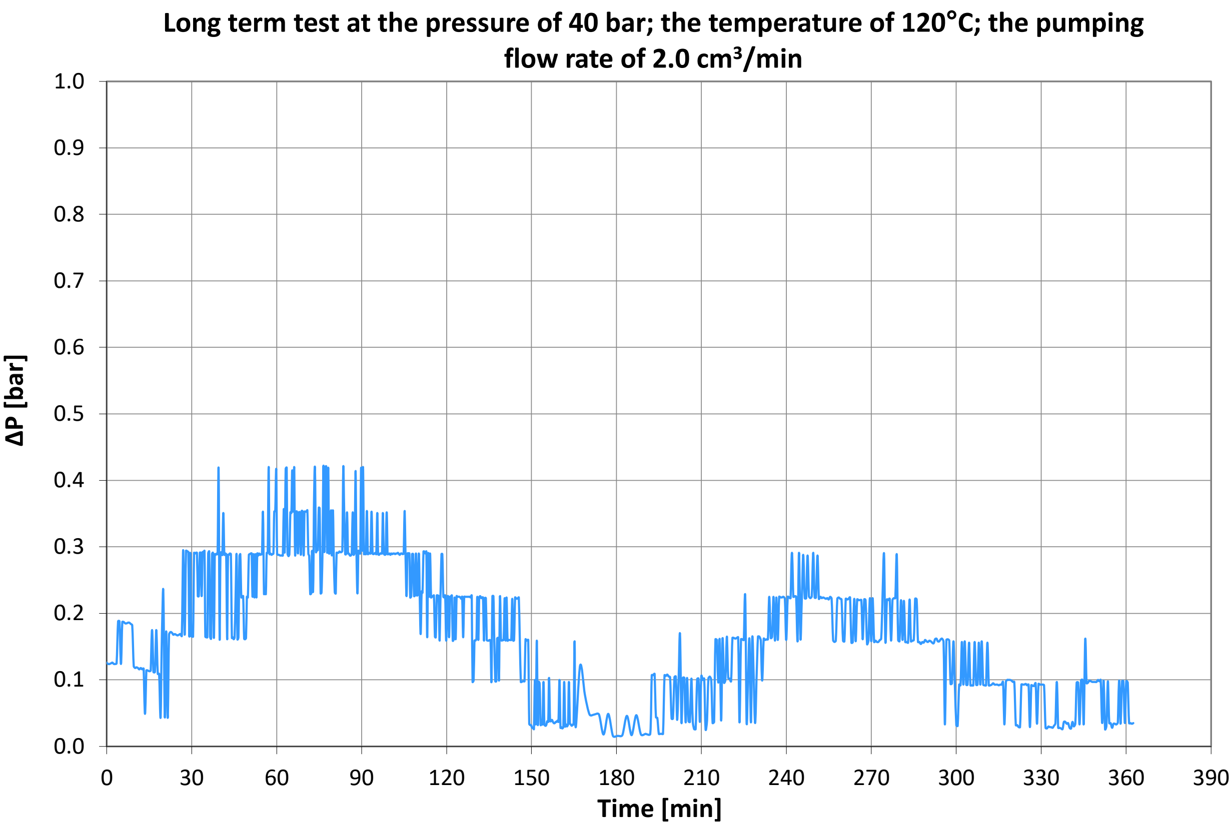

Figure 8. Long-term test of waters "A" and "B" under the following conditions: P = 40 bar, T = 120 °C and q = 2.0 cm^3/min for each of the brine types (measurement error is ±4%).

Table 2. Sediment precipitation in geothermal waters measurement results.

Item	Test Temperature [°C]	Test Pressure [bar]	Pumping Flow Rate [cm^3/min]	Build-Up Start [min]	End of the Test [min]	Differential Pressure (Final) [bar]	Sodium Bicarbonate Concentration [800 g/mL]
1	85	40	2	Brine flow is not blocked			-
2	120	40	2	Brine flow is not blocked			-
3	85	40	1	30.0	32.7	4.897	2
4	85	40	2	20.7	26.9	5.014	2
5	85	18	1	47.7	59.9	4.806	2
6	85	18	2	42.0	54.9	4.570	2
7	120	40	1	10.8	13.7	4.749	2
8	120	40	2	8.3	11.3	4.777	2
9	120	18	1	20.2	40.1	4.712	2
10	120	18	2	15.0	26.1	4.867	2
11	85	40	1	151.1	213.1	4.053	1
12	120	40	1	31.4	42.2	4.663	1
13	120	40	2	26.0	40.9	4.305	1
14	120	18	1	42.0	65.3	4.815	1
15	120	18	2	36.5	67.9	3.304	1
16	120	40	2	Brine flow is not blocked—long-term test (end of the test: after 362.5 min)			

4. Discussion

The main purpose of the work was to check the possibility of using the equipment for PVT tests to determine the conditions of inorganic sediment precipitation in geothermal waters. Geothermal waters drawn directly from boreholes were used in the tests. The first tests were performed using the brines collected on the site. As they did not show a tendency to block the flow, one of them was modified to initiate a precipitation effect. In total, a dozen or so measurements were taken.

The performed tests confirmed the possibility of determining the conditions for the precipitation of inorganic sediments in geothermal waters. The beginning of the flow resistance increase in the tube was taken as the final blocking time of the capillary. As mentioned above, the research was also carried out with the modified water "A". The modification consisted in dissolving a specific mass of sodium bicarbonate in 800 mL of the water. The concentrations used were 1 and 2 g/800 mL, respectively. Sodium bicarbonate was not dissolved in a larger volume of brine to save the test material (limited amount available). Such a volume was however sufficient to carry out the measurement. Depending on the concentration, as well as the pressure and temperature conditions of the test, the capillary blocking times ranged from just a few minutes to several hundred minutes. The variation in the results allowed the authors to determine that the measurements were performed correctly and it is possible to use PVT equipment for testing the precipitation of sediments in geothermal waters.

5. Conclusions

1. The test stand, which was made based on the PVT equipment in place, allows determination of the conditions of precipitation of inorganic sediments in geothermal waters in a dynamic test, following the recommendations set out in NACE TM0197-2010, No. 21228.
2. Figure 5 shows the change in the capillary blocking time depending on the pressure and the liquid flow velocity at a temperature of 120 °C. For this temperature, the measurements were made at pressures of 18 and 40 bar and flows of 1 and 2 cm^3/min for each of the brines. The blocking time of the capillary was between 9–20 min. The graph also shows the different nature of the curves depending on the pressure level. At 40 bar, the pressure will increase more rapidly, which means that deposits will "build up" in the conduit faster, while at 18 bar, the pressure increase is much smoother. This is due to the much slower accumulation of inorganic deposits on the capillary walls.

3. The test series conducted using geothermal waters under the given dynamic test conditions (pressure, temperature, flow rate) confirmed the suitability of using PVT equipment to determine the conditions of precipitation of inorganic sediments in geothermal projects.
4. To eliminate the risk of deposition of solids in geothermal waters, it is necessary to select and introduce appropriate inhibitor/inhibitors.
5. The selection of inhibitors is affected by many factors, including the type and intensity of sediments, the chemical composition of the water and the method of introducing the inhibitor into the circuit.
6. Solid sediments inhibitors, as well as other chemicals used, e.g., paraffin inhibitors, must be periodically adjusted to be adequate for the changing chemistry of the extracted and reinjected brine [30].

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Nomenclature

PVT	**Pressure, Volume, Temperature**—shorthand for name of device for the study of phase changes;
RES	renewable energy source;
NACE	International, a not-for-profit professional organization for the corrosion control industry whose mission is to “[equip] society to protect people, assets and the environment from the adverse effects of corrosion”. NACE International’s membership includes engineers, inspectors, technicians, scientists, business owners, executives, researchers, educators, students and others. The association is organized into four areas in North America and four global areas. NACE International has 142 sections, including 33 student sections worldwide, which sponsor local programs to promote the exchange of corrosion information and education. Among NACE members, the main focus of activities includes cathodic protection, coatings for industry, inspection, corrosion testing and material selection for specific chemical resistance;
pH	quantitative scale of acidity and alkalinity of aqueous solutions of chemical compounds;
mg/L	the number of milligrams dissolved in a liter of solution;
bar	pressure unit;
ΔP (bar)	differential pressure;
cm^3/min	unit of flow rate;
g/800 mL	the amount dissolved in 800 mL of liquid;
P	Pressure;
T	Temperature;
q	Pumping speed;
TBT	Tube Blocking Test.

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