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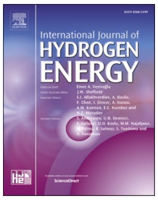

Assessment of the feasibility of hydrogen storage in salt caverns: Implications for purity and contamination pathways for fuel cell mobility

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ABSTRACT

Salt caverns are increasingly considered for large-scale hydrogen storage to support Germany's energy transition and decarbonization goals. This study investigates the feasibility of hydrogen storage in a small salt cavern in Rüdersdorf, Germany, focusing on hydrogen purity after underground storage. Using controlled injection and withdrawal experiments, hydrogen samples were analyzed for contaminants according to EN 17124:2022. The results demonstrate that the hydrogen purity remained high (>99.95 %) after storage, however, nitrogen (up to 450.07 μmol/mol), water (up to 67.95 μmol/mol), and halogenated compounds (up to 0.159 μmol/mol) exceeded the standard's thresholds. Nitrogen contamination is linked to the initial use of a nitrogen blanket during cavern construction and diminished after subsequent injections. Persistent water and halogenated compound concentrations indicate the need for limited purification steps to meet fuel cell mobility requirements. This study provides crucial empirical data on contaminant behavior in salt cavern hydrogen storage, advancing understanding for future large-sale applications.

1. Introduction

The European Union, including Germany, is confronted with significant challenges in the energy sector. The continent is pursuing ambitious climate targets, as set out in the European Green Deal and in national energy policies such as the German "Energiewende" [1–5]. Germany is currently undergoing a significant transformation in its energy sector, driven by the country's ambitious energy transition actions. The objective of this transformation is to limit climate change effects by reducing carbon dioxide (CO_2) emissions with transitioning from fossil fuels to renewable energy sources, including wind and solar energy. However, the generation of renewable energy is subject to fluctuations, which presents a considerable obstacle to the provision of a stable and dependable energy supply [6,7]. In order to address this issue, it is of importance to implement energy storage solutions, with hydrogen (H_2) emerging as a pivotal factor in this context. In addition to its potential as a green fuel for industry and transportation, H_2 represents a promising solution for large-scale energy storage, particularly if produced by electrolysis using surplus renewable energy [5,7–11].

In comparison to alternative long-term and large-scale storage solutions, including e-fuels, methanol and ammonia (NH_3), gaseous H_2 presents a number of significant advantages. Although liquid energy storage solutions possess higher energy densities and are applicable in specific contexts, gaseous H_2 offers higher flexibility, as it cannot only be used as an energy carrier, but also as a raw material for numerous industrial processes, including chemical industry, steel production and refineries [8,11]. Furthermore, it can be more readily integrated into existing gas infrastructures, thereby enhancing its usability as a versatile storage medium [12–17]. This makes it a key factor in the decarbonization of not only the energy sector, but also industry and transport.

It is anticipated that there will be a notable increase in the demand for H_2 in Germany in the forthcoming years. Studies indicate that demand for H_2 is projected to reach 90 to 110 TWh per year by 2030, with industry and the transport sector representing the primary consumers [5,18]. It is therefore essential that storage infrastructure is developed in order to meet this demand while also supporting the expansion of renewable energies. Salt caverns, which are underground cavities typically formed in geological formations from salt deposits, have gained

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considerable attention as potential large-scale (TWh's) storage facilities for H_2 [19–23]. Due to their distinctive properties, including high gas tightness, the utilization of these caverns for the storage of natural gas has been a successful, established and ongoing practice in Germany and across Europe for several decades, showing the technical feasibility and economic viability [22,24–29]. Such storage facilities can be utilized to buffer inter- and intra-seasonal fluctuations in the energy generation and demand, thereby contributing to the overall stabilization of the energy system.

In Germany, cavern storage facilities are currently mainly used to store natural gas. It is conceivable that these existing storage facilities could be converted for the storage of H_2, thus matching the future demand of H_2 and providing a total storage capacity of 30–35 TWh [23, 30]. For Germany is estimated that a capacity of approximately 50–100 TWh of H_2 storage will be required in 2050 to ensure the long-term storage and supply of H_2 for industry and the transport sector [23, 31–33].

Nevertheless, the utilization of H_2 in energy systems presents a number of challenges. While the potential benefits of H_2 storage in salt caverns are obvious, a number of technical challenges have to be addressed. These include the design of compressors and drying systems, measurement equipment (flow and mass), regulation, the quality of H_2 and leak tightness. In particular, the impact of underground H_2 storage on the purity of the stored H_2 needs to be considered. Given that a low level of H_2 purity results in durability issues of the fuel cell and, consequently, reduced long-term viability for use in the transport sector, ensuring the required quality is of importance for applications in fuel cell mobility [34,35]. In order to comply with the demanding quality standards set out in the DIN EN 17124:2022 (EN 17124) and ISO 14687:2019 (ISO 14687) [36,37], which stipulates a minimum purity (hydrogen index) of 99.97 % for H_2 in fuel cell applications for vehicles, it is necessary to utilize high-purity H_2. The quality of the gas produced varies considerably depending on the source of the H_2 [38,39]. "Green" H_2, which is produced by electrolysis using renewable energy sources, is characterized by a high level of purity [39]. In contrast, H_2 produced from fossil sources, such as steam methane reforming of natural gas ("grey" H_2) or coal gasification ("blue" H_2 with carbon capture and storage), often contains impurities such as nitrogen (N_2), hydrocarbons or sulfur compounds and must be purified after production to comply with EN 17124 [38,39]. During the storage period, H_2 of high purity can become contaminated because of interactions with the surrounding geological conditions, including moisture and other trace elements, or the materials used to operate the caverns (e.g. steel, polymer seals) as well as by microbial metabolism.

The presence of potential contaminants, including water (H_2O), oxygen (O_2), halogenated compounds and sulfur compounds, has the potential to affect the performance of fuel cells. Therefore, it is important to gain an understanding of the impact of the storage environment on the purity of H_2 and determine which conditions have to be met to enable H_2 storage while maintaining the H_2 purity.

The objective of this research is twofold: firstly, to assess the feasibility of using salt caverns for the inter- and intra-seasonal storage of high-purity H_2; and secondly, to identify potential contamination pathways that could affect H_2 quality during the storage. The newly constructed salt cavern in Rüdersdorf (Germany) which was specifically designed for H_2 storage, serves as test environment. The findings will be used for optimization of such storage facilities for future applications in the German energy landscape.

2. Materials and methods

2.1. Sampling of hydrogen at salt caverns

The on-site sampling of gases from the cavern was conducted using a commercially available Swagelok sampling system, coupled with SilcoNert® 2000 coated Swagelok sampling cylinders in order to minimize the interaction of contaminants with the stainless steel of the cylinder (Fig. 1) [34,40]. Moreover, a markedly augmented cylinder wall thickness is employed to facilitate sampling at up to 17.5 MPa (the maximum cavern pressure). The entire sampling system is situated in close proximity to the cavern head within the inlet and outlet piping, thus enabling the cylinders to be filled with direct cavern pressure. The cylinders have a volume of 0.5 L each. In order to analyze the H_2 with the mass spectrometer, a minimum of 40 L of H_2 at atmospheric pressure is required. Therefore, a sampling pressure of 8 MPa is necessary. At lower cavern pressures, two cylinders were combined for the analysis to have enough sample volume for the entire measurement. The cylinders were transported in accordance with the relevant Transportable Pressure Equipment Directive (TPED) approvals.

In order to obtain representative samples, the method of pressure swing purging was initially employed, followed by continuous purging with the gas to be analyzed. In order to achieve this, the sampling cylinder was affixed to the apparatus and linked via quick-connectors. Any excess H_2 was vented via a connected blow-out stack as part of the above-ground infrastructure of the cavern site. The cylinder was initially filled with H_2 via cavern pressure, up to a maximum of 17 MPa, with the outlet closed. It was then depressurized to atmospheric pressure, with the inlet closed and the outlet open. Subsequently, the cylinder was subjected to an additional purge of H_2 from the cavern for 60 s, this time with the outlet open. The sampling procedure was concluded by closing the outlet to allow for the build-up of the sample pressure in the cylinder. Following the release of the pipe pressure of the Swagelok sampling system, the cylinders were securely stored in their cases and transported to the laboratory for analysis.

In order to obtain data relevant to the current operating status of the cavern, extended flushing of H_2 was required depending on the flow rate of the injection or withdrawal of H_2. This ensured that H_2 that had already been in the pipes for some time was not sampled.

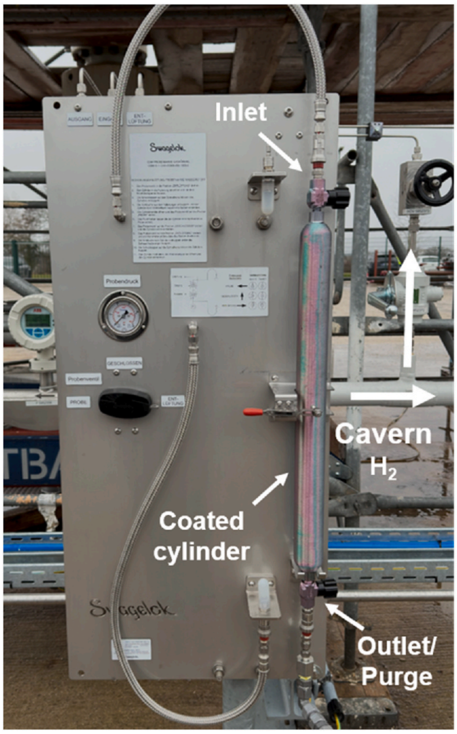

Fig. 1. Sampling system combined with coated 0.5 L sampling cylinder for sampling of hydrogen at cavern pressure (max. 17.5 MPa) at a hydrogen storing cavern with inlet (top) and outlet/purge (bottom).

2.2. Analysis of hydrogen with mass spectrometry

The analysis of contaminants in H_2 is carried out by mass spectrometry (MS) using the CombiSense instrument from V&F, Austria. The methodology underlying this work has been described in a previous study in detail elsewhere [34]. In the aforementioned study, an analytical approach was developed for the identification of contaminations in H_2 samples in accordance with EN 17124. This approach entailed the utilization of Ion Molecule Reaction Mass Spectrometry (IMR-MS). A key element of the study was the calibration of the IMR-MS and the selection of the requisite gases. The methodologies devised for contamination-free sampling at hydrogen refueling stations and analysis can be partially adapted to this study for salt caverns, even if an upgraded configuration for the verification of H_2 quality according to EN 17124 is employed in this context. In this study, the specific technique of IMR-MS has been complemented by Electron Impact Mass Spectrometry (EI-MS) in the CombiSense instrument. With IMR-MS and a low ionization energy of 10–14 eV, molecules can be analyzed with low detection limits (described in Ref. [34]). At the same time, concentrations of molecules can be determined with EI-MS where a higher ionization energy is necessary (e.g. N_2), or because of high concentrations (higher μmol/mol-range and lower percent-range). With EI-MS, ionization is conducted at 70 eV and separation is performed using a quadrupole mass filter. This provides an additional mass range of 1–100 u (atomic mass unit) with a resolution of <1 u. The molecules and fragments are registered by a fast pulse counter. The described components of the H_2 analyzer are shown in a flowchart in Fig. 2. This technique provides reliable results (accuracy $< \pm 2$ %, reproducibility $< \pm 3$ %) with a fast response time (T90 < 20 ms for each fragment, depending on the measurement task) with particularly low detection limits, as required by EN 17124.

The gases to be analyzed in the cavern samples are listed in Table 1 together with the detection limits resulting from the calibration and compared with the given limits of the standards. A two-point calibration was performed daily to calibrate the analyzer. High purity H_2, produced on-site from H_2 5.0 (Linde, Germany) using a palladium membrane (Entegris MegaTorr® PS7-PD05-08), resulting in H_2 9.0 quality (hydrogen index >99.9999999 %) according to the data sheet, was used as the background and to calculate the limit of detection (LOD) and limit of quantification (LOQ). According to ISO 21087:2019 (ISO 21087) [41], the LOD was calculated using the 3-fold standard deviation by repeating ten analyses of H_2 9.0. For the LOQ, the standard deviation was multiplied by factors depending on the expected concentrations of the analytes. For concentrations according to EN 17124 greater than or equal to 1 μmol/mol a factor of 10 is used, for values between 10 nmol/mol and 1 μmol/mol a factor of 5 and for values smaller than 10 nmol/mol a factor of 3. The latter results in corresponding values for LOQ and LOD.

Table 1
Contaminants and thresholds listed in the comprehensively convergent standards ISO 14687:2019 and DIN EN 17124:2022 with corresponding limits of detection (LOD) and limits of quantification (LOQ) of the analyzer resulting from 2-point calibration with calibration gases and inert gas (H_2 9.0) according to ISO 21087.

Contaminants	ISO 14687/EN 17124 [36,37] (μmol/mol)	Limit of detection (LOD) analyzer (μmol/mol)	Limit of quantification (LOQ) analyzer (μmol/mol)
Water (H_2O)	5	1.570	5.233
Total non-methane hydrocarbons (NMHC)[a]	2	0.008	0.013
Methane (CH_4)	100	0.090	0.300
Oxygen (O_2)	5	0.075	0.250
Helium (He)	300	0.180	0.600
Nitrogen (N_2)	300	0.078	0.260
Argon (Ar)	300	0.019	0.063
Carbon dioxide (CO_2)	2	0.021	0.070
Carbon monoxide (CO)	0.2	Not measured	Not measured
Total sulfur (TS)[b]	0.004	0.001	0.002
Formaldehyde (HCHO)	0.2	0.005	0.008
Formic acid (HCOOH)	0.2	0.002	0.003
Ammonia (NH_3)	0.1	0.001	0.002
Total halogenated (TH)[c]	0.05	0.014	0.023

[a] With Xylene (m-, p-, o-) (C_8H_{10}), Toluene ($C_6H_5CH_3$), Benzene (C_6H_6), Ethylene (C_2H_4), Acetylene (C_2H_2),n-Pentane (C_5H_{12}), Butane (C_4H_{10}), Ethane (C_2H_6).

[b] With Carbon disulfide (CS_2), Hydrogen sulfide (H_2S), Methanethiol (CH_3SH), Ethanethiol (C_2H_5SH), Sulfur dioxide (SO_2), Carbonyl sulfide (COS).

[c] With Tribromomethane ($CHBr_3$), Tetrachloroethylene (C_2Cl_4), 1,2-Dichloroethane ($C_2H_4Cl_2$), Trichloroethylene (C_2HCl_3), Trichloromethane ($CHCl_3$), Dichloromethane (CH_2Cl_2), Hydrogen chloride (HCl).

H_2 5.0 was used as purge gas during standby operation. In all cases, calibration gas standards were obtained from Siad, Germany using background gas H_2 6.0 or, for formaldehyde (HCHO) and formic acid (HCOOH), N_2 6.0. In addition to what was already described in Ref. [34], the gases N_2, argon (Ar), helium (He), as well as more detailed sum parameters for hydrocarbons, sulfur compounds and halogenated compounds were added to the measurement procedure. For the analyses of H_2 samples, reliable results are obtained after 15–30 min at a flow rate

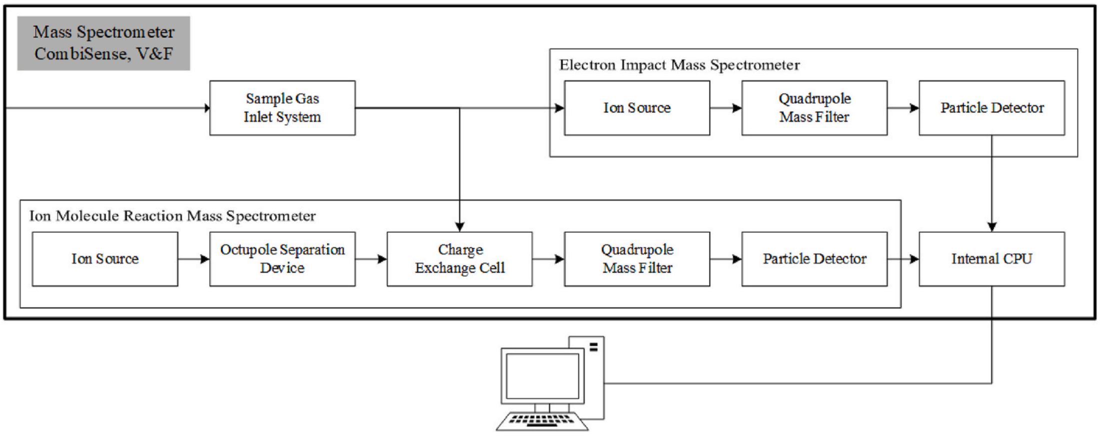

Fig. 2. Scheme of the mass spectrometer CombiSense, V&F based on ion molecule reaction (IMR-MS) and electron impact (EI-MS) mass spectrometry for the laboratory analysis of contaminants in hydrogen samples.

of 1 L per minute.

For the determination of the total sulfur content (TS), carbon disulfide (CS_2), hydrogen sulfide (H_2S), methanethiol (CH_3SH), ethanethiol (C_2H_5SH), sulfur dioxide (SO_2) and carbonyl sulfide (COS) were selected for analysis as representative sulfur-containing impurities because of their good volatility in calibration gases and a certain probability of occurrence from previous measurements. The parameter total halogenated compounds (TH) includes all halogens and halogenated inorganic and organic compounds. For this, tribromomethane ($CHBr_3$), tetrachloroethylene (C_2Cl_4), 1,2-dichloroethane ($C_2H_4Cl_2$), trichloroethylene (C_2HCl_3), trichloromethane ($CHCl_3$), dichloromethane (CH_2Cl_2) and hydrogen chloride (HCl) were analyzed as representative impurities based on previous studies [36,42]. Xylene (meta-, para-, ortho-) (C_8H_{10}), toluene (C_7H_8), benzene (C_6H_6), ethylene (C_2H_4), acetylene (C_2H_2), n-pentane (C_5H_{12}), butane (C_4H_{10}) and ethane (C_2H_6) were used for the analysis of total non-methane hydrocarbons (NMHC). To specify the concentration of the respective sum parameters, the individual contributing components are weighted in accordance with EN 17124. For example, in the case of C_2Cl_4 this results in a 4-fold weighting for the parameter TH and in a 7-fold weighting of C_7H_8 for NMHC (C_1-equivalent). H_2O was calibrated only once by the manufacturer of the CombiSense device, due to the lack of availability of specific H_2O concentrations in calibration gas standards, and was therefore not recalibrated in the laboratory prior to each measurement.

As shown in Ref. [34], despite the absence of fragmentation of the molecules, interferences are present due to similar and/or equal *m/z* ratios. While minor interferences, such as those measured of O_2 on C_2H_6, C_4H_{10} on CO_2, or $C_2H_4Cl_2$ on C_2H_5SH, can be accurately determined during calibration, the influence of N_2 on carbon monoxide (CO) is so significant that for CO, despite the precise analysis of N_2 concentration with EI-MS, no linearity can be observed with the 2-point calibration, and consequently, CO cannot be quantified reliably. For this reason, CO is not included in the measurements.

ISO 21087 [41] defines the validation of equipment and methodology for analyzing H_2 in the laboratory. It classifies suitability criteria for selectivity, LOD and LOQ, working ranges, bias, precision, measurement uncertainty and robustness. The aim is to enable laboratories to prove that the method used is suitable for the analysis of contaminants in H_2. Due to the use of high precision IMR-MS and EI-MS technology, and the sampling and analysis method developed, validation for research purposes is not mandatory [34]. For each analyzed cylinder the mean value was calculated from five measurements. The error calculation of the reported measurements is based on ISO 21087 [41] by calculating the relative combined total uncertainty ($k = 2$) including the contributions of the individual uncertainties, i.e. standard deviation of the analytical values, calibration (calibration gases), and accuracy and precision of the instrument. Analyses of simulated H_2 samples with unknown concentrations and interlaboratory comparisons with real H_2 samples have demonstrated that the underlying methodology and instrument parameters allow validation based on ISO 21087.

3. Results and discussion

3.1. Storage of hydrogen in a salt cavern in Rüdersdorf, Germany

A salt cavern for H_2 storage formed in a salt pillow of the saliniferous formation Zechstein by solution mining with fresh water in a gas cavern storage field (EWE GASSPEICHER, Germany) was constructed in Rüdersdorf, Germany. After completion, the cavern was echometrically measured resulting in a height of 20.6 m (at a depth between 1216.0 and 1236.6 m) and a diameter of 6.6 m (at a depth of 1230.0 m). The volume of the cavern is 458.2 m^3. After the first injection of H_2, a brine volume of 10.6 m^3 was determined. With the purpose to serve as a test environment for fuel cell grade H_2 storage according to EN 17124, the volume of this cavern is much smaller compared to other natural gas caverns (50,000 to 700,000 m^3) and the cavern neck comparably long with 180 m [43]. The composition of the salt deposit in this region and at this depth is largely dominated by sodium chloride. Geological studies have identified a working pressure range of approximately 6–17 MPa. At the lowest possible working pressure, residual gas needs to remain in the cavern, the so-called cushion gas, and is therefore not useable during injection and withdrawal phases. After the necessary waiting time after solution mining (setting of the rock), an average temperature for the gas of 58 °C was determined at this depth of the cavern. As this study is concerned with the quality of the stored H_2, details of geology and engineering of the cavern will not be discussed here, but can be found elsewhere [19–23,30,43].

This cavern was newly built, and therefore has never been used to store natural gas or crude oil. N_2 was used as a blanket during the solution mining process to control the shape of the cavern and prevent the upper salt layer from dissolving, rather than diesel or other hydrocarbons [44]. This has fundamental advantages in terms of preventing salt cavern contamination, e.g. by minimizing hydrocarbon contamination. Thus, this cavern offers a basis for providing H_2 in fuel cell grade quality after storage.

Subsequently to the solution mining of the cavern and preliminary successful gas tightness tests, the initial H_2 injection into the cavern was conducted (displacement of the brine with H_2). In order to meet the fuel cell grade (99.97 %) described in EN 17124, it would be necessary to apply H_2 5.0 (99.999 %) for the injection process (H_2 4.0 or comparable is not available for delivery). However, the delivery of H_2 5.0 would not have been possible for this project due to technical and logistical challenges. Therefore, H_2 with a quality of 3.0 (99.9 %, Linde) was injected. By definition, the quality of H_2 3.0 would be already too low for the hydrogen index described in EN 17124 (Table 1) and therefore for fuel cell mobility. However, preliminary analyses have shown that the H_2 3.0 is usually of a much higher quality (>99.99 %) and therefore actually meets the requirements of EN 17124. 19 trailer transports were necessary for the initial injection of H_2, so that a total of approximately 63,000 Nm^3 of H_2 was stored at a cavern pressure of approximately 17 MPa. In order to be able to make statements about cavern-induced, i.e. building and operation of the cavern, contamination routes after the subsequent withdrawal of the H_2, eight of 19 selected trailers delivering H_2 were sampled during the first injection process and two of nine trailers during the second injection phase. Subsequently, the H_2 quality was determined in the laboratory (Table 2). With regard to the interpretation of the measured concentrations, it should be noted that < LOD represents a value below the respective LOD, while < LOQ indicates a value between the respective LOD and LOQ as indicated in Table 1. The respective hydrogen index (total hydrogen contamination) was calculated by accumulating the individual values actually measured. If the measured value of the contamination was below the LOD or LOQ, the respective latter value from Table 1 was used instead.

Overall, except of one contamination value from trailer 3, all other values of each sampled trailer of the first injection phase were below EN 17124 thresholds (Table 2). In general, for the sum parameter of halogenated contaminants, values below the LOD of 0.014 μmol/mol were found, but for trailer 3, a concentration of 0.052 ± 0.014 was slightly above the limit of 0.05 μmol/mol. Concentrations of water were found to be around the LOD and LOQ. As a result of contact between H_2 stored in the cavern and brine and the cavern sump during the storage phase, significant contamination with H_2O is to be expected during withdrawal phase. The low measured values for H_2O are therefore a useful benchmark for further assessment of the suitability of the cavern for fuel cell mobility at this point. In addition, O_2 concentrations ranging from 0.295 ± 0.040 to 1.107 ± 0.118 μmol/mol were measured, which are below the threshold of 5 μmol/mol, but should be considered for later analysis during extraction. Concentrations of He and Ar were measured in the range of 50 and 4 μmol/mol, respectively, which is typical for H_2 produced via the steam reforming process [39,45]. No change of these values is expected during the storage process. For N_2, low concentrations up to a maximum of 17.62 ± 1.56 μmol/mol were found, which were

Table 2
Overview of the quality of hydrogen sampled from gas delivering trailers before it was stored in the salt cavern.

Contaminants	EN 17124 [36] (μmol/mol)	Measured concentrations of contaminants in hydrogen (μmol/mol)							
		First injection phase						Second injection phase	
		Trailer 1	Trailer 2	Trailer 3	Trailer 4	Trailer 5	Trailer 6	Trailer 1	Trailer 2
Water (H_2O)	5	< LOD	< LOD	< LOQ	< LOD	< LOD	< LOQ	< LOQ	< LOQ
Total non-methane hydrocarbons (NMHC)	2	< LOD	< LOD	0.093 ± 0.018	0.020 ± 0.004	< LOQ	< LOQ	< LOQ	< LOQ
Methane (CH_4)	100	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Oxygen (O_2)	5	0.750 ± 0.073	0.295 ± 0.040	1.107 ± 0.118	0.340 ± 0.042	0.827 ± 0.115	0.922 ± 0.110	0.951 ± 0.208	0.543 ± 0.281
Helium (He)	300	47.57 ± 4.43	42.17 ± 3.93	45.31 ± 4.22	42.12 ± 3.94	42.48 ± 3.96	45.20 ± 4.21	49.36 ± 5.26	45.69 ± 4.79
Nitrogen (N_2)	300	2.08 ± 0.21	1.24 ± 0.17	17.62 ± 1.56	1.46 ± 0.15	2.86 ± 0.39	2.64 ± 0.24	2.94 ± 0.65	1.03 ± 0.43
Argon (Ar)	300	4.59 ± 0.40	4.64 ± 0.41	4.36 ± 0.38	4.22 ± 0.37	3.29 ± 0.29	2.50 ± 0.22	0.88 ± 0.09	0.47 ± 0.18
Carbon dioxide (CO_2)	2	< LOQ	< LOD	0.108 ± 0.011	< LOD	< LOD	< LOD	< LOD	< LOD
Total sulfur (TS)	0.004	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Formaldehyde (HCHO)	0.2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Formic acid (HCOOH)	0.2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Ammonia (NH_3)	0.1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Total halogenated (TH)	0.05	< LOD	< LOD	0.052 ± 0.014	< LOD	< LOD	< LOD	0.056 ± 0.019	< LOD
Total non-hydrogen gases	300	56.75 ± 5.11	50.06 ± 4.55	73.98 ± 6.32	49.86 ± 4.51	51.17 ± 4.76	56.85 ± 4.78	59.55 ± 6.23	53.11 ± 5.68
Hydrogen index (%)	99.97	99.994	99.995	99.993	99.995	99.995	99.994	99.994	99.995

therefore not critical regarding the limit value of 300 μmol/mol. Overall, a hydrogen index of >99.99 % (quality 4.0) was determined, which largely meets the requirements of EN 17124 and therefore appears to be a suitable starting point for investigating the influence of H_2 storage in caverns on H_2 purity.

Table 3
Overview of the hydrogen quality after storage in the salt cavern during the first withdrawal phase (storage of H_2 for 16 weeks) and the second withdrawal phase (storage of H_2 for 10 weeks) at different cavern pressures.

Contaminants	EN 17124 [36] (μmol/mol)	Measured concentrations of contaminants in hydrogen (μmol/mol)										
		First withdrawal phase with sampling at different cavern pressures								Second withdrawal phase with sampling at different cavern pressures		
		16.0 MPa	15.8 MPa	15.0 MPa	13.0 MPa	12.8 MPa	8.1 MPa	7.4 MPa	6.9 MPa	15.4 MPa	9.4 MPa	7.2 MPa
Water (H_2O)	5	< LOQ	< LOD	< LOQ	9.01 ± 3.70	33.35 ± 13.70	46.02 ± 18.83	67.95 ± 27.87	64.09 ± 26.28	21.70 ± 9.07	54.61 ± 22.37	72.25 ± 29.92
Total non-methane hydrocarbons (NMHC)	2	< LOQ	0.040 ± 0.016	0.155 ± 0.050	0.084 ± 0.026	0.076 ± 0.017	0.093 ± 0.025	0.090 ± 0.049	0.111 ± 0.028	0.081 ± 0.022	0.055 ± 0.029	0.055 ± 0.021
Methane (CH_4)	100	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Oxygen (O_2)	5	0.418 ± 0.171	0.405 ± 0.103	0.608 ± 0.098	0.992 ± 0.197	0.417 ± 0.056	2.190 ± 0.304	1.692 ± 0.350	1.330 ± 0.486	< LOQ	0.909 ± 0.220	1.448 ± 0.517
Helium (He)	300	38.38 ± 3.96	40.86 ± 4.18	39.60 ± 4.02	39.49 ± 5.03	40.13 ± 4.25	38.60 ± 4.54	40.04 ± 4.27	42.49 ± 5.08	43.53 ± 4.69	42.97 ± 4.67	42.20 ± 5.24
Nitrogen (N_2)	300	196.15 ± 17.12	222.42 ± 19.42	450.07 ± 39.56	429.84 ± 37.46	411.61 ± 35.89	303.75 ± 26.49	298.97 ± 26.07	296.52 ± 25.93	117.23 ± 10.24	147.78 ± 12.90	141.51 ± 13.55
Argon (Ar)	300	3.23 ± 0.43	3.43 ± 0.32	3.93 ± 0.56	4.23 ± 0.41	3.50 ± 0.42	4.02 ± 0.40	4.24 ± 0.46	4.18 ± 0.63	1.65 ± 0.32	3.05 ± 0.33	2.26 ± 0.71
Carbon dioxide (CO_2)	2	< LOQ	< LOQ	0.277 ± 0.025	0.118 ± 0.015	0.102 ± 0.017	0.267 ± 0.027	0.303 ± 0.029	0.328 ± 0.032	0.255 ± 0.027	1.711 ± 0.161	1.475 ± 0.139
Total sulfur (TS)	0.004	< LOD	< LOD	0.003 ± 0.001	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	0.003 ± 0.001	< LOQ	< LOQ
Formaldehyde (HCHO)	0.2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Formic acid (HCOOH)	0.2	< LOD	< LOD	< LOQ	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
Ammonia (NH_3)	0.1	< LOD	< LOD	< LOD	< LOD	< LOD	0.066 ± 0.008	0.101 ± 0.012	0.123 ± 0.016	< LOQ	0.027 ± 0.004	0.025 ± 0.003
Total halogenated (TH)	0.05	< LOD	< LOD	0.042 ± 0.012	0.063 ± 0.017	0.070 ± 0.025	0.075 ± 0.030	0.159 ± 0.019	0.105 ± 0.030	< LOQ	0.092 ± 0.017	0.113 ± 0.041
Total non-hydrogen gases	300	243.61 ± 21.68	268.91 ± 24.04	500.02 ± 44.33	483.93 ± 46.86	489.36 ± 54.38	395.18 ± 50.65	413.64 ± 59.13	409.38 ± 58.51	184.82 ± 24.37	251.30 ± 40.70	261.44 ± 50.14
Hydrogen index (%)	99.97	99.976	99.973	99.950	99.952	99.951	99.960	99.959	99.959	99.982	99.975	99.974

3.2. Influence of the cavern storage to hydrogen purity

In order to demonstrate the suitability of salt caverns as H_2 storage facilities, several operating conditions were tested, including different periods of holding the H_2 in the cavern without pressure changes and different rates of pressure change during withdrawal. In total, two different withdrawal phases were considered in this study. A total of eleven samples were taken and analyzed in the laboratory. The results are presented in Table 3.

In the first withdrawal phase, the H_2 pressure in the cavern was reduced from approximately 17 to 6 MPa. The rate of pressure change was 0.5–0.6 MPa per day. The storage duration before withdrawal was 16 weeks. Eight H_2 samples were taken and analyzed from 16 to 6.9 MPa during the 14-day withdrawal period. Before discussing individual concentrations of the contaminants, it can be anticipated that the overall quality of the withdrawn H_2 was generally very high, but that individual contaminants exceeded the required limits for fuel cell mobility based on EN 17124.

The purity of the withdrawn H_2 was affected by storage in the cavern, with the contamination of H_2O, N_2, NH_3 and TH occasionally exceeding their respective limits set in EN 17124 at certain cavern pressures. The concentrations of all other contaminants were within the individual limits of the standard. The high N_2 concentrations (from 196.15 ± 17.12 to 450.07 ± 39.56 μmol/mol) in the withdrawn H_2 originate from the use of N_2 as a blanket during cavity formation and the need to control the solution mining process. As this process is completed, it is assumed that the concentrations of N_2 will decrease over the number of injections and withdrawals. Indications for that are found in the second withdrawal phase, as will be discussed below. Furthermore, the H_2O concentrations exceed the EN 17124 limit. Due to the wet underground conditions and the presence of a brine sump, there is direct contact with moisture. It is therefore likely that the H_2 is enriched with H_2O over the storage time of 16 weeks. It is remarkable that the concentration increases particularly at low pressures, with a concentration below the LOQ measured from 16 to 15 MPa and up to 67.95 ± 27.87 μmol/mol from 8.1 to 6.9 MPa. As there is more moisture in the deeper layers of the cavern (sump), higher concentrations of H_2O can be detected at lower pressures.

At pressures below 7.4 MPa, the NH_3 concentrations of 0.101 ± 0.012 and 0.123 ± 0.016 μmol/mol are slightly above the limit specified in EN 17124. In addition, TH exceeds the limit of 0.05 μmol/mol by up to 0.159 ± 0.019 μmol/mol due to the presence of trichloroethene and trichloromethane in concentrations of up to 0.052 ± 0.006 and 0.098 ± 0.012 μmol/mol, respectively. Two likely options are assumed for these three contaminants. Firstly, as the cavern was filled with tap and pond/process water during solution mining, contamination of H_2 by contact with the brine components might be possible. NH_3 is more commonly found in tap water due to environmental influences and fertilizers [46]. Halogenated compounds can be found in tap water additionally. These contaminants could have entered the tap water as degradation products from industrial waste water or disinfection during aftertreatment [47,48]. However, the higher concentrations of NH_3 were found only at lower pressures, where contact with potentially contaminated brine is particularly intense. The second possibility as source of contamination, less likely for NH_3 but more likely for halogenated contaminants, is the delivered and injected H_2 from the trailers. One of the six sampled trailers delivering H_2 showed higher concentrations of exactly trichloroethene and trichloromethane (Table 2, Trailer 3). Since not all of the 19 H_2 delivering trailers were sampled and H_2 3.0 was ordered according to the certificate, further and higher concentrations of trichloroethene and trichloromethane within some of the trailers cannot be excluded. As the concentrations exceeding the limit for NH_3 were only found at certain pressures (8.1–6.9 MPa), it is possible that some specific contaminated trailers were not sampled prior to injection.

The concentrations of O_2 after withdrawal, regardless of the gas pressure in the cavern, were found to be in the same range as in the samples taken from the trailers. This confirms that the cavern is highly gas-tight and that no air from atmosphere in significant amounts has permeated in the cavern. Thus, the N_2 concentrations mentioned are not related to air leaks. Due to the use of various polymers and connectors for steel pipes etc., a certain level of hydrocarbon contamination was expected in the samples. However, it is encouraging to note that only low concentrations of NMHC were found despite the analysis of various groups of substances. Furthermore, the He concentration that was already found in the samples of the H_2 delivering trailers, was also detected in the samples from the cavern in the same order of magnitude as beforehand. Apart of these findings, there were no other relevant contaminations in the H_2 in the first phase of withdrawal. Overall, the H_2 was contaminated with a maximum of 500.02 ± 44.33 μmol/mol of non-hydrogen gases, resulting in a minimum hydrogen index of 99.950 %.

After the first withdrawal phase, the cavern was again filled with approximately 39,000 Nm^3 H_2 3.0 (Linde) via nine trailer deliveries from 6 to 17 MPa, analogous to the previous initial gas injection. The gas analysis of two sampled trailers gives evidence for a high H_2 quality ($\geq$ 99.99 %) comparable to the results of the initial gas injection (Table 2). Again, one of both samples showed a slightly higher concentration of TH (0.056 ± 0.019 μmol/mol) compared to the threshold value of 0.05 μmol/mol in EN 17124 while all other compounds were below the limiting values.

The storage duration without withdrawal was 10 weeks. During the second withdrawal phase, the pressure was similar to the first phase reduced from approximately 17 to 6 MPa. In contrast to the first withdrawal phase, a higher rate of pressure change of 7 MPa per day was applied. Within the two days of withdrawal, three samples were taken in a cavern pressure range of 15.4 to 7.2 MPa. Overall, the hydrogen index was in a similar order of magnitude to the first withdrawal phase (Table 3). However, for N_2, significantly lower concentrations of 117.23 ± 10.24 to 147.78 ± 12.90 μmol/mol were measured during the second withdrawal phase due to the diluting effect of the second injection phase with pure H_2 (Table 2). This observation confirms the statement that the first injected H_2 was considerably contaminated by the N_2 blanket used for the solution mining and that there are no other relevant sources of N_2. Further dilution is expected for subsequent injection and withdrawal phases, eliminating N_2 as a relevant contaminant for this cavern.

In contrast to the first withdrawal phase, concentrations of NH_3 were measured well below the 0.1 μmol/mol EN 17124 limit, with a maximum of 0.027 ± 0.004 μmol/mol. For the first withdrawal, the relevant concentrations exceeding the limit were measured at 7.4 and 6.9 MPa. During the second withdrawal, less NH_3 was identified at 7.2 MPa with 0.025 ± 0.003 μmol/mol. According to the previous explanation in the section about N_2, dilution could be a reason for the lower NH_3 concentrations. Since no NH_3 was found in all of the sampled trailers (Table 2) and there was no further solution mining or use of tap water, the dilution mechanism via injection of pure H_2 during the second injection phase is a probable reason for the decline in NH_3 concentration. Further investigation should include continuous monitoring of this contamination in order to detect any exceedance of the limit at an early stage.

At a high cavern pressure of 15.4 MPa, a value below the LOQ (0.023 μmol/mol) was measured for TH. However, as in the first withdrawal phase, it increased to 0.113 ± 0.041 μmol/mol at 7.2 MPa, which is above the specified EN 17124 limit of 0.05 μmol/mol. Once again, trichloromethane and trichloroethene were responsible for the limit being exceeded. Compared to the first withdrawal phase, the values are very similar, so that a contamination of the H_2 over the entire pressure range is probable. Thus, in contrast to NH_3, no dilution of TH could be detected. As exactly the same two contaminants, trichloromethane and trichloroethene, were found in one of the two sampled H_2-supplying trailers during the second injection phase, it can be assumed that many more trailers were contaminated with these contaminants. The assumption of contamination via tap water can therefore be ruled out at least as the only cause of contamination with halogenated compounds. As with NH_3, it would be advisable to take further samples during the continued use of the cavern.

For H_2O, comparable concentrations of 21.70 ± 9.07 (15.4 MPa) to 72.25 ± 29.92 μmol/mol (7.2 MPa) were detected as in the first withdrawal phase. The values are slightly higher for the second phase at high pressure (15.4 MPa). A possible explanation for this is the cavern temperature of approximately 60 °C. Due to low molecular mass and the resulting high thermal conductivity of 0.186 $W\ m^{-1}\ K^{-1}$ [49], the H_2 quickly adopts the ambient temperature during withdrawal, so that condensation was possible at and directly behind the cavern head during withdrawal. Consequently, with a much higher withdrawal rate, as in the second phase, a higher concentration is reasonable. In any case, concentrations above the threshold are to be expected after salt cavern storage. If the H_2 shall subsequently be used for fuel cell mobility, a drying step must always be provided to ensure compliance with EN 17124 and the water concentration must be monitored [50,51].

While low concentrations of CO_2 up to a maximum of 0.303 ± 0.029 μmol/mol were found during the first withdrawal phase, relevant concentrations not exceeding the limit (2 μmol/mol) up to a maximum of 1.711 ± 0.161 μmol/mol at 9.4 MPa were found during the second withdrawal phase. Contamination from H_2 delivering trailers during both injection phases, which were not sampled in this study, is again considered possible. Contamination from the air can be excluded due to the low concentrations of N_2 and O_2. Compared to the first withdrawal phase, the concentration for O_2 remains in a very similar range.

With the exception of the He concentration caused by using “grey” H_2 mentioned in the section on initial gas injection, there are no other relevant contaminations of the H_2 in the second withdrawal phase. Overall, the H_2 was contaminated with a maximum of 261.44 ± 50.14 μmol/mol of non-hydrogen gases, resulting in a minimum hydrogen index of 99.974 %.

Comparing the two withdrawal phases, the overall results are striking. During storage in the salt cavern, the quality of the H_2 is impaired so that it cannot be used directly for fuel cell mobility without purification. However, the contamination is rather small and the overall quality of the H_2 remains very high at $\geq$ 99.950 %. In particular, critical components such as sulfur, methane (CH_4), CO_2, O_2 and hydrocarbons were detected at very low levels below the limits specified in EN 17124. The contaminants N_2 and NH_3 found in this study will presumably dilute during further operation of the cavern with more injection and withdrawal phases and therefore do not represent a risk factor. The only remaining challenges are halogenated compounds and, above all, H_2O. However, the rise in the amount of H_2O in H_2 was expected after storage and can be minimized by drying steps such as absorption processes.

The results for impurities in H_2 caused by cavern storage, for example TS and CH_4 concentrations, which were mostly below the LOQ, demonstrate that no significant amounts of metabolites from H_2-consuming microorganisms could be detected. This indicates that there was no activity of microorganisms at levels relevant to influence H_2 quality for inter- and intra-seasonal storage. However, microbial activities might require initially a certain latency to adapt to the environment. A study by Dohrmann et al. [52] was conducted at the same cavern site in Rüdersdorf to characterize the microbial communities and to predict their potential to consume and contaminate stored H_2. The objective was to identify the microorganisms initially present in the fresh water used for solution mining (pond water) and after 345 days in the brine from of the cavern. Although typical H_2-consuming (oxidizing) microorganisms were found in the pond water, they were quickly deactivated upon injection to the cavern, presumably due to the drastic change in conditions (high salinity, temperature, and pressure). In contrast to that, in the brine sample, a community dominated by highly-adapted halophiles was found after one year in the cavern. It is important to note, that laboratory incubation experiments with this cavern brine could not identify microbial activities potentially affecting H_2 quality, e.g. by sulfate reduction, methanogenesis, or acetogenesis [52].

In addition to microorganisms, reactions between salt rock and H_2 via dissolution and/or precipitation in salt caverns might affect H_2 purity. Salt caverns are formed within salt domes, which are primarily composed of halite (NaCl). Halite is considered chemically inert, but impurities such as quartz, anhydrite, gypsum, dolomite, pyrite, and calcite can be present in interlayers [53,54]. For instance, anhydrite/gypsum or pyrite might react with H_2 and brine to H_2S [53–55]. In this study, the cavern was created in the Zechstein formation [56]. The salt pillow in Rüdersdorf is largely composed of halite (>99.5 %) with minor amounts of sylvite (KCl). Studies have shown that in halite-dominated salt domes, geochemical reactions induced by H_2 are of minor significance and kinetically hindered [53,54]. Consequently, the impact of these reactions on H_2 purity during inter- and intra-seasonal storage is anticipated to be limited, as was shown by the respective H_2 purity after storage. Storage periods substantially longer than those considered are out of the scope of this study since they are not relevant for the investigated cavern.

The authors would like to point out that the contaminations and corresponding measured concentrations apply to the H_2 stored in this specific cavern in Rüdersdorf, Germany. As the geology and nature of the cavern will vary in shape, depth, width, height, pressure and temperature, the general validity of the statements made here need to be verified [22,28,57]. This would require sampling and analysis of different caverns. As the use of salt caverns for H_2 storage is still in its early stages, a number of follow-up studies will be carried out. It will be important to determine what the purpose of the cavern is. The often discussed standard EN 17124 with a hydrogen index of 99.97 % (Grade D) will not necessarily be the desired level for each cavern. In studies [58–60], an industrial quality of 98 % (Grade A) or 99.5 % is often considered sufficient for pipelines and therefore also for caverns. The H_2O concentrations determined for this cavern are within the acceptable range for Grade A, but further consideration should be given to the amount of H_2O to prevent corrosion of piping and related steel components [24]. For fuel cell mobility, purification to Grade D independent of storage is therefore recommended to limit costs for more widespread applications.

4. Summary and outlook

The construction and operation of the H_2 storage cavern in Rüdersdorf, Germany, is an important step in demonstrating the viability of underground salt caverns for H_2 storage, particularly for applications requiring high-purity H_2, such as fuel cell mobility. The study focused on a small-scale 458.2 m^3 cavern built specifically for controlled experiments to assess how inter- and intra-seasonal underground storage affects H_2 purity. With a working pressure range of 6–17 MPa and a sodium chloride-dominated geological composition, this cavern provided a unique test environment. The use of N_2 as a blanket during the solution mining process, created conditions well suited to studying H_2 storage without contamination from previous industrial uses such as natural gas or crude oil storage.

The primary objective of this study was to evaluate the feasibility of storing H_2 in salt caverns and to assess the effect of this storage on the purity of the H_2, particularly in relation to the EN 17124 standard, which requires a minimum hydrogen index of 99.97 %. Despite the use of H_2 with grade 3.0 (99.9 %) for the initial injection due to logistical constraints, the analyses showed that the delivered and fed in H_2 had a higher quality (>99.99 %), approaching the requirements described by EN 17124. Laboratory analyses of H_2 samples from the injection and withdrawal phases provided insight into potential contamination pathways.

Key findings show that while the overall quality of the stored H_2 remained high (99.950 % during the first and 99.974 % during the second withdrawal phase), certain contaminants exceeded the limits specified in EN 17124. In particular, N_2 concentrations reached up to

450.07 ± 39.56 μmol/mol in the first withdrawal phase, mainly due to the N_2 blanket used during cavern creation. However, N_2 concentrations were significantly reduced during the second withdrawal phase due to the diluting effect of pure H_2 injected in the second injection phase, supporting the assumption that the N_2 contamination was related to the initial N_2 blanket rather than air leakages or cavern fractures and resulting in a less significant long-term risk. H_2O contamination was another significant finding for both withdrawal phases, with comparable concentrations increasing to 72.25 ± 29.92 μmol/mol at lower cavern pressures due to contact with the brine sump. This confirms that moisture is a persistent challenge in salt caverns. As expected, this study highlights the need for a drying step for H_2 intended for fuel cell applications after storage in such caverns.

In addition, halogenated compounds, in particular trichloromethane and trichloroethene, were found at concentrations above the limiting threshold of EN 17124 (up to 0.159 ± 0.019 μmol/mol) during the first and second withdrawal phase. This contamination was probably introduced by H_2 delivering trailers during the injection phases, highlighting the need for more stringent controls and sampling of H_2 from these trailers, as only eight of 28 incoming trailers were analyzed in this study. Interestingly, NH_3 concentrations exceeded the EN 17124 limit (up to 0.123 ± 0.016 μmol/mol) at lower pressures only within the first withdrawal phase, possibly due to contaminants in the water used for brining or environmental influences. As in the case of N_2, the dilution effect of the second injection phase with pure H_2 results in significantly lower concentrations measured in the second withdrawal phase with a maximum of 0.027 ± 0.004 μmol/mol.

Overall, the results of this study indicate that H_2 storage for fuel cell mobility in salt caverns is feasible. However, purification processes will be necessary to maintain the high H_2 purity required for fuel cell mobility described by EN 17124. After storage, no significant increases in the concentrations for O_2 or hydrocarbons were measured, confirming the caverns structural integrity. However, halogenated compounds and water remain challenges to be addressed in future research. The results are specific to the geological conditions and construction techniques of the Rüdersdorf cavern, and broader validation in different geological settings (e.g. with high microbial activity) is required to generalize the results. This study is an important milestone in the evaluation of the continued use of salt caverns as part of the future energy system, as these tests did not identify any effects that would fundamentally limit the use of salt caverns for H_2 storage in general. Future research should focus on investigating larger caverns, optimizing pressure and withdrawal rates, evaluating the microbial activity, and implementing cost-effective purification techniques. As H_2 storage becomes increasingly important for renewable energy integration and fuel cell mobility, understanding and mitigating contamination risks in underground storage will be critical to the long-term success of these technologies. The challenge of achieving a balance between preventing contamination, for example through the use of high-purity H_2 for injection, the selection of appropriate materials and the holistic purification of the H_2 after withdrawal, while optimizing the cost and efficiency of the cavern, requires further research and development. While EN 17124 purity standards are critical for certain fuel cell applications, a lower hydrogen index (e.g. 98 %, Grade A) may be sufficient for industrial applications, reducing the need for extensive purification directly at the cavern after storage. However, for fuel cell applications, a purification step is likely to remain necessary to meet the high H_2 quality standards required for mobility solutions.

CRediT authorship contribution statement

Holger Janßen: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Conceptualization. **Barbara Satola:** Writing – review & editing, Project administration. **Michael Kroener:** Writing – review & editing, Supervision, Project administration, Funding acquisition. **Alexander Dyck:** Writing – review & editing, Supervision. **Martin Vehse:** Writing – review & editing, Supervision. **Michael Wark:** Writing – review & editing, Supervision. **Carsten Agert:** Writing – review & editing, Supervision.

Statement

During the preparation of this work the authors used DeepL in order to improve the text quality. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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