International Journal of Hydrogen Energy 77 (2024) 520–525

Contents lists available at ScienceDirect

International Journal of Hydrogen Energy

journal homepage: www.elsevier.com/locate/he

Feasibility of induction heating for the dehydrogenation of liquid organic hydrogen carriers

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ARTICLE INFO

Handling Editor: Ramazan Solmaz

Keywords:
Induction heating
Dehydrogenation
Hydrogen production
Perhydro-benzyltoluene

ABSTRACT

Electromagnetic induction heating could eliminate the limitations associated with conventional heating methods for endothermic dehydrogenation reactions of liquid organic hydrogen carriers—it may offer faster heating rates, while directing the heat from the core of the inductively active materials to the catalyst and the surrounding fluid. A 5 wt% Pt/γ-Al_2O_3 prepared by wet impregnation, was used as a wash-coat to produce an inductively active catalyst in which stainless steel (SS) pellets were used. Different configurations were considered, for comparison: SS pellets alone, Pt/γ-Al_2O_3 coated SS pellets, a mixture of 5 wt% Pt/γ-Al_2O_3 pellets and SS pellets. An induction heating set-up for the catalytic dehydrogenation of perhydro-benzyltoluene (H12-BT) was built (use was made of an inductive coil covering a glass batch reactor. Increasing the n(Pt)/n(H12-BT) ratio lowered the catalyst productivity ($gH_2gPt^{-1}min^{-1}$) by 7.4%, but enhanced the H12-BT conversion and selectivity towards the product benzyltoluene (H0-BT). Remarkably, there are no by-products in either the liquid or gas phases at high conversion (>90%).

1. Introduction

The hydrogen produced from renewable and sustainable energy sources plays a key role in enabling the decarbonisation of various industries—including, but not limited to, transportation, manufacturing, energy production, chemicals and petrochemicals [1]. Although hydrogen is considered a promising alternative to fossil fuels, one of the significant challenges that hinder its more widespread adoption is the efficiency and costs associated with its storage and distribution [2]. While liquid organic hydrogen carrier (LOHC) technology offers many advantages for the large-scale storage and distribution of hydrogen [3], one of the challenges associated with this technology is the high energy demand of the endothermic dehydrogenation reaction [4].

To date, heating sources for the dehydrogenation of LOHCs include electrical heating mantles [5], jacketed reactors using heat transfer fluids [6], gas burners [7], exhaust heat from solid oxide fuel cells [8], gas turbines [9] and waste heat from industrial plants [10]. An assessment of the existing integration of LOHC systems with various heat sources reveals significant differences in terms of efficiencies. For example, the integration of a LOHC system with a polymer electrolyte membrane fuel cell, where a dehydrogenation reactor is electrically heated, exhibited an efficiency of 20% [11]. For a LOHC–solid oxide fuel cell integrated system, where the exhaust heat is utilised for the dehydrogenation reaction, an efficiency of 38% was reported [11]. Moreover, an LOHC reactor system heated by a porous hydrogen burner, where a share of the hydrogen produced is used for heating a dehydrogenation reactor, exhibited an efficiency of 28% [11,12]. The most conventional heating method for LOHC dehydrogenation is based on electrical elements, however, this method has several drawbacks. Wang et al. report that induction heating using radio frequency induction overcomes non-uniform temperature distribution within the reactor, as well as the slow heating and cooling rates associated with conventional heating methods [13].

However, the suitability of induction heating for the dehydrogenation reactions of conventional LOHC molecules has not been fully explored yet. A few years ago, Hart et al. [14] compared the effects of conventional heating and induction heating for the dehydrogenation of tetralin, wherein stainless steel (SS) pellets were used as a substrate. Results indicated that inductive heating led to improved

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https://doi.org/10.1016/j.ijhydene.2024.06.229
Received 24 April 2024; Received in revised form 13 June 2024; Accepted 16 June 2024
Available online 19 June 2024

Fig. 1. Stainless steel pellets used in the dehydrogenation of H12-BT by induction heating method.

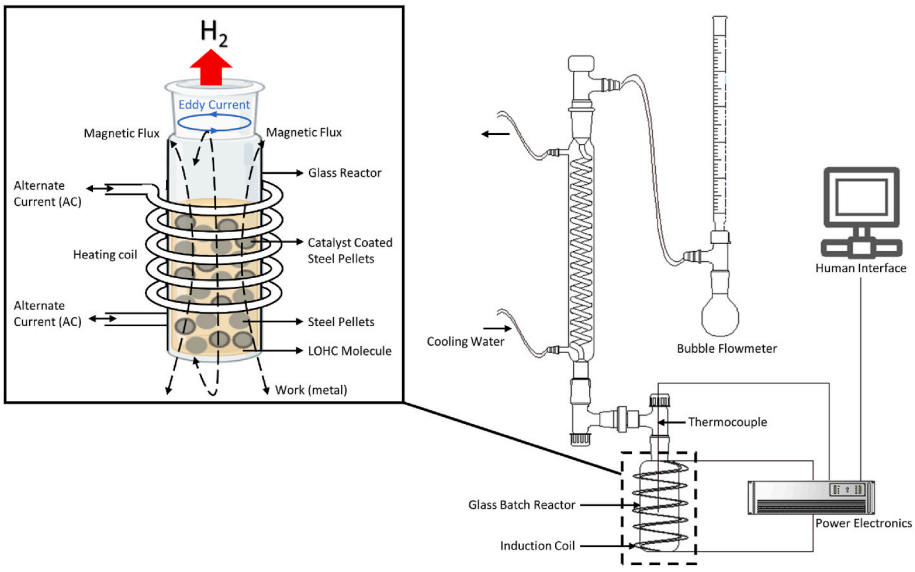

Fig. 2. Experimental setup of an inductively heated batch type reactor for the catalytic dehydrogenation of H12-BT.

dehydrogenation productivity for Pt/γ-Al_2O_3-coated SS pellets due to the effective heat transfer offered by the SS pellets to the catalyst and bulk fluid. However, while the heat transfer process from the source to the catalyst is compromised when using electrical heating [14], electromagnetic induction heating offers more efficient direct and targeted heat delivery, faster heating and cooling rates, reduced heat loss to the environment and increased energy efficiencies, all while maintaining consistent reaction temperatures [13,15].

In this study, we investigate the feasibility of induction heating for the dehydrogenation of perhydro-benzyltoluene (H12-BT). To accomplish this objective, the following tasks were carried out. Firstly, we modified an off-the-shelf induction heating module to meet the specifications of the glass batch reactor and developed an experimental dehydrogenation set-up with a Raspberry Pi-based controller. Secondly, we coated the inductively active material (SS) with Pt/γ-Al_2O_3 catalyst. Thirdly, we optimised the induction heating system for the desired dehydrogenation temperature, to avoid extreme overheating. Lastly, we conducted LOHC dehydrogenation performance tests to compare the effectiveness of different catalyst configurations.

2. Materials and methods

2.1. Catalyst preparation

The 5 wt% Pt/γ-Al_2O_3 catalyst pellets were prepared by the wet impregnation method as described in literature [16]. Use was made of γ-Al_2O_3 pellets (Clariant, South Africa) and chloroplatinic acid precursor. The γ-Al_2O_3 support material had a surface area of 186 m^2g^{-1}, a pore volume of 0.6 cm^3g^{-1} and a pore diameter of 12 nm [5]. To prepare the chloroplatinic acid, a Pt sponge (>99% purity; Impala Platinum Limited, Johannesburg, South Africa) was dissolved in aqua regia. Using inductively coupled plasma optical emission spectrometry; the Pt content of the impregnated support was determined to be 38%.

To prepare the catalyst coated SS pellets, the spherical SS pellets (4.5 mm; Guerrilla, South Africa) were etched with 5 M nitric acid (ACE Chemicals, Johannesburg solution to improve the surface roughness for better adhesion (see Fig. 1.) Polyvinyl alcohol (MW 22000, high purity grade) was supplied by VWR International (Leuwen, Belgium). Milli-Q water was used to prepare a 1 wt% binder solution. A slurry was then prepared by mixing grounded 5 wt% Pt/Al_2O_3 and the binder solution into which the pre-treated SS pellets were dipped, followed by drying. The dip coating was repeated until the desired coating had been obtained. The catalyst treatment gases were supplied by Afrox (Johannesburg, South Africa): N_2 (99.999%) and H_2 (99.999%). Fig. 1 shows the SS pellets: untreated, etched in acid, and coated with Pt/γ-Al_2O_3.

2.2. Induction heating: experimental set-up

The catalytic dehydrogenation of H12-BT was carried out using the induction heating set-up as shown in Fig. 2. The set-up consisted of a two-port 50 ml batch reactor made of glass, with a thermocouple insert and a cooling condenser connected to a soap bubble flow meter.

The heating set-up comprised a power supply and induction heating module interfaced with a Raspberry Pi IO board for parameter input and data recording. A 250-W power supply that converts 220 V AC to 12 V DC provided the power to the IO board and the induction heating

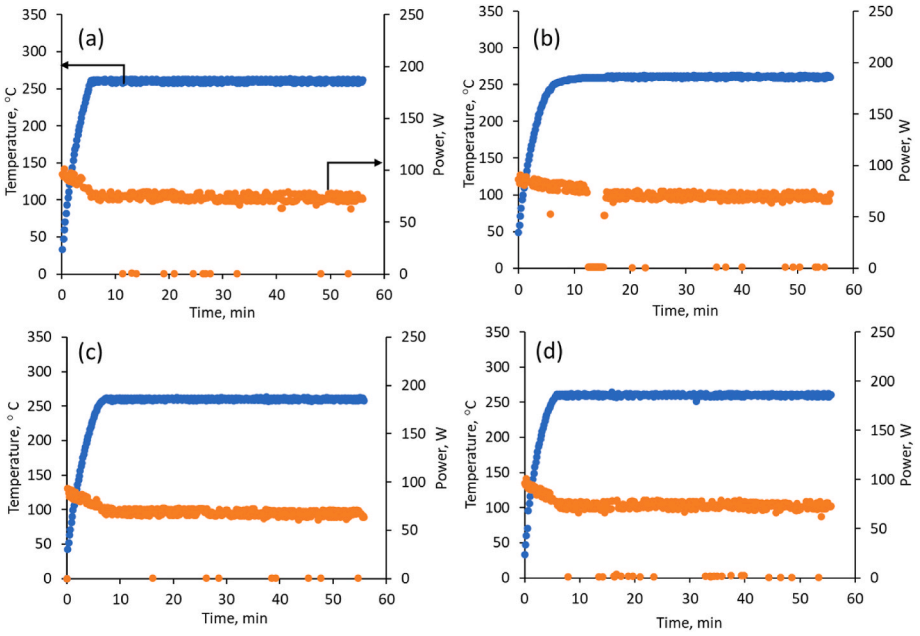

Fig. 3. Comparison of the temperature and power consumption for various configurations: (a) uncoated SS pellets, (b) Pt/γ-Al_2O_3 + SS, $n_{Pt}/n_{H12\text{-}BT}$ = 0.52 mol%, (c) Pt/γ-Al_2O_3 coated SS $n_{Pt}/n_{H12\text{-}BT}$ = 0.11 mol%, (d) Pt/γ-Al_2O_3 + SS, $n_{Pt}/n_{H12\text{-}BT}$ = 0.11 mol%. Process conditions: 50 ml batch reactor, T = 260 °C, reaction time = 55 min, $V_{H12\text{-}BT}$ = 10 ml.

module. The glass batch reactor was placed inside the copper coil of the induction module, as shown in Fig. 2. Inductively active materials (SS pellets) were added to the glass reactor to facilitate heating through eddy currents induced by the AC magnetic field generated by the heating coil. Use was made of an induction coil, powered by a high-efficiency zero-voltage switching printed circuit board, and copper tubing with water cooling to protect the electronics from overheating. At a resonant frequency of ±200 kHz, the coil effectively heated the SS pellets through eddy currents (±17 A_{rms}), while minimising power losses in the driver circuit. The turns of the coil were determined by the thickness of the tubing and the height of the section filled with SS pellets in the glass reactor. The tight winding of the coil's inner diameter around the glass reactor ensured a strong magnetic field coupling to the SS pellets.

2.3. Evaluation of induction heating for dehydrogenation of H12-BT

Prior to the commencement of induction heating, a glass reactor was filled with 10 ml of H12-BT (99% degree of hydrogenation), followed by the addition of the inductively active material (SS pellets) to the height of the induction coil. A thermocouple was placed vertically in the centre of the reactor to measure and control the heating, while the hydrogen flow was measured using a bubble soap meter. The temperature of the induction heating was controlled by the software interface. Three reactor packing configurations were investigated to elucidate the effect of Pt/γ-Al_2O_3 catalyst pellets and catalyst coating on SS pellets for the inductively heated dehydrogenation reaction. Three systems were tested: i) SS pellets (4.5 mm)—as the control experiment, ii) Pt/γ-Al_2O_3 catalyst pellets mixed with SS pellets (Pt/γ-Al_2O_3 + SS), where 2 M ratios were investigated, namely, $n_{Pt}/n_{H12\text{-}BT}$ = 0.11 mol% and 0.52 mol%, and iii) SS pellets coated with a Pt/γ-Al_2O_3 coating (Pt/γ-Al_2O_3 coated SS) at 0.11 mol%.

2.4. Analysis of the H12-BT reaction mixture

The reaction mixture of H12-BT was analysed using gas chromatography coupled to a single quadrupole mass spectrometer system (GC-SQ-MS) (Scion, Goes, The Netherlands), as previously described [16]. The GC-SQ-MS was equipped with a CP-8400 autosampler and a GS-Tek GsBP-50 + MS column (30 m length, 0.25 mm internal diameter, 0.25 μm film thickness), and helium (99.999%) was used as the carrier gas at a flow rate of 1 ml/min. Hexane (Sigma-Aldrich Germany) was used to dilute samples of the reaction mixture (1:100 wt/wt). The injector was set at 300 °C and 1 μl aliquot samples were injected at a split ratio of 1:100. The oven temperature was increased from 70 °C (held for 2 min) to 160 °C at a rate of 40 °C/min, followed by a further increase to 300 °C at 2.5 °C/min (then held for 20 min). The transfer line and the source temperature were maintained at 250 °C and 200 °C, respectively. The electron energy was maintained at −70 eV.

3. Results and discussion

During testing of the induction heating, technical challenges were evident when using only SS pellets immersed in H12-BT. For example, the AC magnetic field interfered with the thermocouple measurements, particularly at >200 °C. To address this, the induction heater circuit was then automated to periodically switch off every 2 s, for 0.1 s, to obtain more accurate temperature measurements, compensating for the decreasing magnetic permeability of the steel pellets when approaching the Curie temperature (720–750 °C; 316 SS) [17]. This is a critical point, beyond which a material loses its magnetic properties, with a reduction in the efficiency of induction heating.

Fig. 3 shows the induction heating temperature and power consumption when using uncoated SS pellets, SS pellets mixed with catalyst coated SS pellets, and SS pellets mixed with Pt/γAl_2O_3 pellets. In the case of the SS pellets without catalyst, an average power consumption of 73 W was achieved when a temperature of 260 °C was maintained for approximately 50 min (Fig. 3a). In the case of the mixed pellets (Pt/γ-Al_2O_3 + SS pellets) at a catalyst to H12-BT ratio of 0.52 mol%, 66 W was consumed for the dehydrogenation of H12-BT at 260 °C (Fig. 3b), whereas at a lower molar ratio of 0.11 mol%, in the case of the Pt/γ-Al_2O_3 catalyst coated SS pellets, 68 W was required to maintain a dehydrogenation temperature of 260 °C (Fig. 3c). Finally, the Pt/γ-Al_2O_3 pellets mixed with uncoated SS pellets (Pt/γ-Al_2O_3 coated SS at 0.11 mol%) required 67 W for the dehydrogenation of H12-BT (Fig. 3d).

The power consumption for all the dehydrogenation tests using Pt/γ-Al_2O_3 + SS was lower compared to when only SS pellets were used.

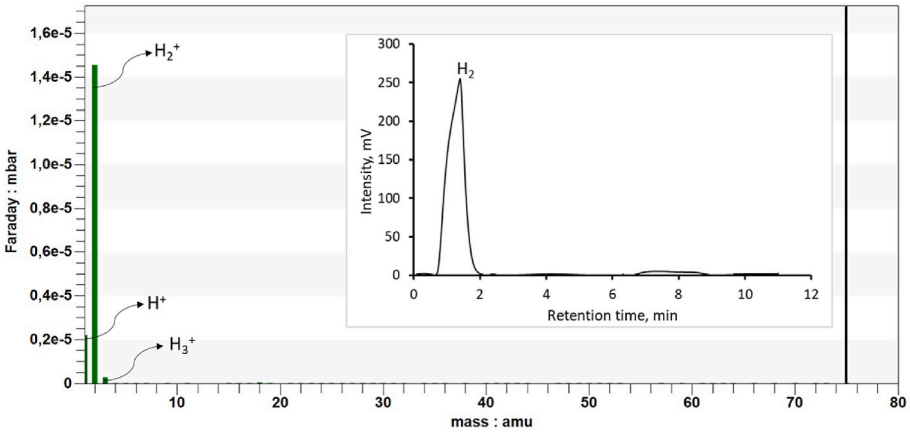

Fig. 4. Mass spectrum and chromatogram (insert) of gas produced during dehydrogenation of H12-BT. Pt/γ-Al_2O_3 + SS (mix): $n_{Pt}/n_{H12\text{-}BT}$ = 0.52 mol %, T = 260 °C.

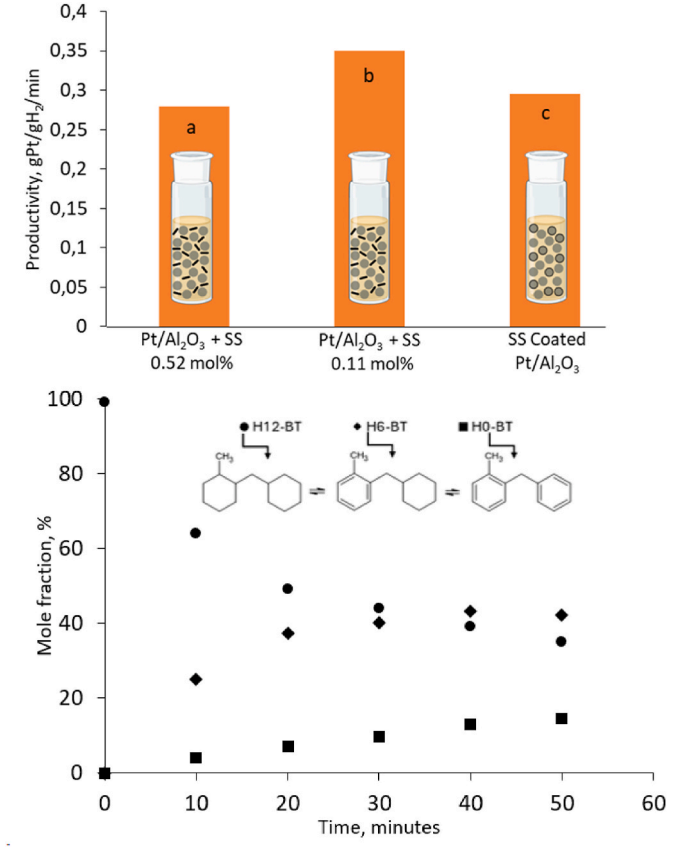

Fig. 5. Top: Comparison of catalyst productivity for the dehydrogenation of H12-BT, using an induction heating batch reactor, for three different catalyst configurations: a) Pt/γ-Al_2O_3 + SS (0.52 mol%), b) Pt/γ-Al_2O_3 + SS (0.11 mol %), c) Pt/γ-Al_2O_3 coated SS (0.11 mol%). Bottom: Product distribution over reaction time for the dehydrogenation of H12-BT using Pt/γ-Al_2O_3 + SS (0.52 mol%) at 260 °C (a).

This observation suggests that while the core material (SS) is responsible for the induction heating due to its electrical conductivity and magnetic permeability, the coating and Pt/γ-Al_2O_3 catalyst pellets could interfere with how the magnetic field interacts with the SS pellets.

To validate the suitability of induction heating for the dehydrogenation of H12-BT, liquid Samples were taken at 10-min intervals, while the flow rate was recorded manually using a bubble flow meter. The gas, collected using a sample bag, was analysed using a Hiden HPR-20 R&D Specialist Gas Analysis System (Hiden, UK). Using a MASsoft Bar scan with Faraday detector, A clear hydrogen peak (m/z = 2; >99.9%), without any impurities, was observed, see Fig. 4. Moreover, SRI-8610C GC (USA) with thermal conductivity detector, shown only the hydrogen peak and a purity of >99%, see Fig. 4 insert. The catalyst efficiency was calculated in terms of the amount of hydrogen produced per gram of Pt and time ($gH_2gPt^{-1}min^{-1}$) [16]. See Fig. 5. The uncoated SS pellets mixed with Pt/γ-Al_2O_3 catalyst pellets ($n_{Pt}/n_{H12\text{-}BT}$ = 0.11 mol%) exhibited the highest productivity of 0.35 $gH_2gP^{-1}min^{-1}$, followed by the catalyst coated SS pellets ($n_{Pt}/n_{H12\text{-}BT}$ = 0.11 mol%) and the uncoated SS pellets mixed with catalyst pellets ($n_{Pt}/n_{H12\text{-}BT}$ = 0.52 mol%) at 0.29 and 0.27 $gH_2gPt^{-1}min^{-1}$, respectively. In the absence of catalyst, no hydrogen production was observed. The high productivity achieved for Pt/Al_2O_3+stainless steel can be attribute to the absence of PVA binder on the catalyst pellets. Strauch et al., reported on the productivity in the range between 2 and 4.3 $gH_2gPt^{-1}min^{-1}$ using bimetallic platinum rhenium catalyst on alumina for dehydrogenation of H12-BT [18]. Reactive distillation was employed for dehydrogenation of H12-BT at 260 °C, using Pt/Al_2O_3 catalyst and the authors achieved a productivity of 0.48 $gH_2gPt^{-1}min^{-1}$ [19]. Alconada and Barrio incorporated Co, Mo and Mn oxides into Pt/Al_2O_3 for dehydrogenation of H12-BT using a batch reactor and the productivities obtained are for Pt, PtCo, PtMo and PtMn: 1.4, 2, 3.5 and 1.7 $gH_2gPt^{-1}min^{-1}$, respectively [20]. The productivities reported in literature are higher than those reported in this work. However, metal doping, bimetallic catalyst, metal loading, textural properties of the support material as well as metal to LOHC ratio, have an influence on productivity.

The low productivity obtained for Pt/Al_2O_3 coated is because PVA was used as binder for adhesion and this can coat the active sites of the catalyst, physically blocking reactants from accessing these sites, thus reducing the catalyst's effectiveness. Furthermore, while PVA provide

Table 1
Catalyst performance data for the dehydrogenation of H12-BT using induction heating. Process conditions: 50 ml batch reactor, T = 260 °C, reaction time = 55 min, $V_{H12\text{-}BT}$ = 10 ml.

Catalyst	$n_{Pt}/n_{H12\text{-}BT}$ (mol%)	Productivity ($gH_2gPt^{-1}min^{-1}$)	Degree of Dehydrogenation (%)	Conversion (%)	Selectivity (%)
Pt/γ-Al_2O_3 + SS (mix)	0.11	0.35	16	6	3
Pt/γ-Al_2O_3 + SS (mix)	0.52	0.27	45	65	18
Pt/γ-Al_2O_3 coated SS	0.11	0.29	20	15	4

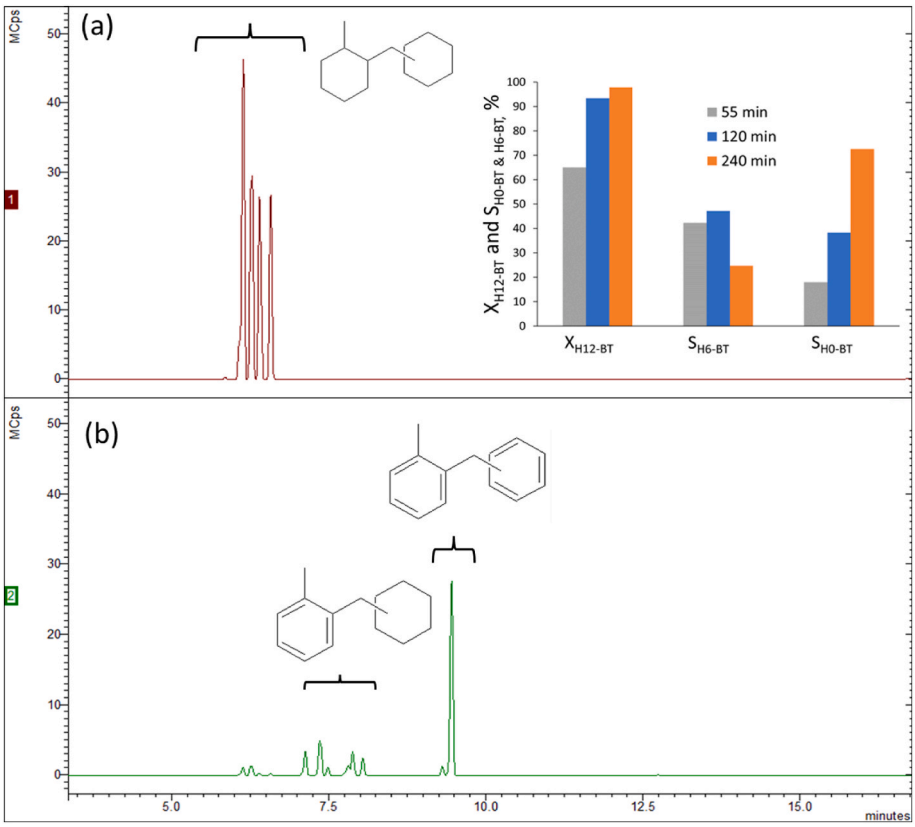

Fig. 6. GC-SQ-MS Total Ion Chromatogram showing H12-BT before reaction (a) and product distribution after 4h of dehydrogenation reaction (b). Insert: H12-BT conversion and selectivity to H6-BT and H0-BT. Pt/γ-Al_2O_3 + SS (mix): n_{Pt}/n_{H12-BT} = 0.52 mol %, T = 260 °C.

structural adhesion it can also potentially alter the pore structure and surface area, which are critical for catalytic activity.

Liquid samples from Pt/γ-Al_2O_3 + SS (0.52 mol%) were drawn from the reactor and analysed using GC-SQ-MS to determine the composition of the product: H12-BT, H6-BT and H0-BT were present (Fig. 5). As expected, the mole fraction of the reactant (H12-BT) decreased with increasing time, leading to the formation of the intermediate H6-BT and the product H0-BT [20]. Within the 50-min reaction time, the H6-BT increased more rapidly than the H0-BT. The concentration of H6-BT decreased during the last 40–50 min, with a subsequent increase in H0-BT. This confirmed the stepwise dehydrogenation reaction pathway reported in literature, where H12-BT converts to H0-BT via H6-BT [20].

Table 1 presents catalyst performance data for the dehydrogenation of H12-BT using induction heating. The critical role of the Pt catalyst/H12-BT molar ratio on the efficiency of the dehydrogenation process is highlighted. A higher molar ratio led to an increased degree of dehydrogenation conversion of H12-BT and selectivity towards the desired dehydrogenated product (H0-BT), although at a reduced productivity. This trade-off suggests that optimising the Pt concentration is crucial for the full exploitation of the hydrogen storage capacity of the H12-BT as well as for maximising the catalyst efficiency. Moreover, a decrease in the concentration of Pt by almost 80% (79%) resulted in a 7.4% change in productivity for Pt/γ-Al_2O_3-coated SS pellets, where an improved productivity implies that less catalyst is required to achieve an increased amount of product, thus making the dehydrogenation process more cost effective.

Furthermore, the configuration of the catalyst, i.e., whether mixed with or coated on SS, has an impact on the reaction. Coating the catalyst

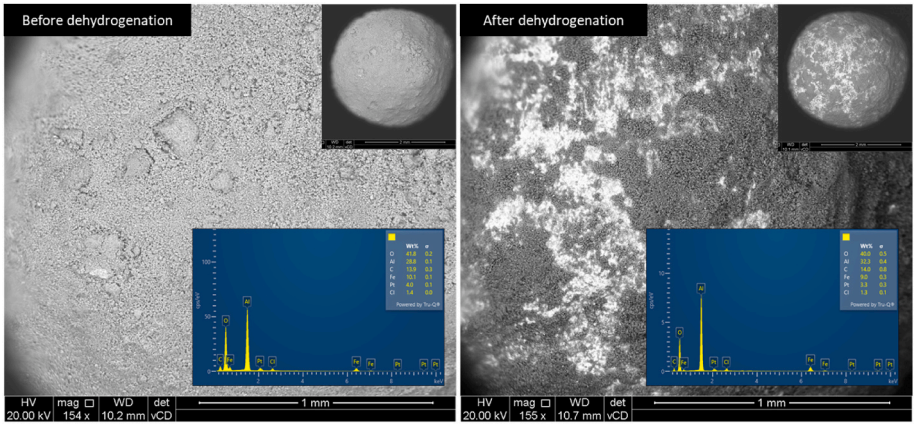

Fig. 7. SEM-EDX images of Pt/γ-Al_2O_3 coated SS pellets, before and after dehydrogenation.

on SS improves the performance slightly compared to mixing it at the same platinum to H12-BT ratio, thus indicating that catalyst–support interactions also play a role in the reaction efficiency. Although the primary objective of this study was to explore induction heating for the dehydrogenation of H12-BT, it is important to align this with performance targets in future research work. Desirable targets for the dehydrogenation of LOHCs include the following: productivity >3 $gH_2/g_{catalyst}$/min, energy demand <6 kWh/kg-H_2, conversion >90% and selectivity >99.8% [21]. Upon comparing the data in Table 1 with the desirable targets for the dehydrogenation of LOHCs it is evident that none of the catalysts considered here achieve a conversion rate that is >90% nor a selectivity target that is >99.8%. The Pt/γ-Al_2O_3 + SS (mix) catalyst with nPt/nH12-BT of 0.11 mol% exhibits the highest selectivity: 18%. While none of the catalysts meet the productivity target (i.e., exceeding 3 gH_2/gPt/min), the highest observed productivity is 0.35 $gH_2gPt^{-1}min^{-1}$ for the first Pt/γ-Al_2O_3 + SS (mix) catalyst at 0.11 mol%. Despite falling short of desired catalyst targets, this study demonstrates the feasibility of induction heating for H12-BT dehydrogenation, with future research focusing on optimization to meet efficiency benchmarks. As shown in Figs. 4 and 6, no by-product observed in both gas and liquid phases at prolonged reaction time (4h) and conversion of 98%. However, some literature studies reported on the formation of methylfluorene as a by-product, resulting from dehydrogenative cyclization as a side reaction [20]. The SEM-EDX in Fig. 7 shows the detachment of the catalyst after the dehydrogenation reaction. This is a challenge that needs to be addressed by improving the either the binder formulation or use of alternative binders and applying different coating methods.

4. Conclusion

The implementation of automated control in the induction heater circuit, which turns off the system for short intervals, successfully mitigated the interference of the AC magnetic field on thermocouple measurements. Induction heating for the dehydrogenation of H12-BT at 260 °C was confirmed by GC-SQ-MS analysis—there was a decrease in H12-BT forming first H6-BT and ultimately H0-BT. For the dehydrogenation of H12-BT, both the quantity of Pt relative to the substrate and the physical configuration of the catalyst system are crucial for optimising the reaction for higher conversion and selectivity for dehydrogenated products. The catalyst adhesion needs to be improved to avoid the detachment observed after dehydrogenation reaction. It is remarkable that, no by-products were observed in both gaseous and liquid phases. Using this preliminary data, we recommend that future studies should include investigating the balance between Pt concentration and catalyst configuration, towards further enhancing the efficiency of this reaction.

CRediT authorship contribution statement

Danae Ford: Writing – original draft, Data curation. **Gert Kruger:** Writing – review & editing, Methodology, Conceptualization. **Rudaviro Garidzirai:** Data curation. **Phillimon Modisha:** Validation, Supervision, Data curation, Conceptualization. **Henning Krieg:** Writing – review & editing, Supervision. **Dmitri Bessarabov:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

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.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

Acknowledgements

North-West University (Potchefstroom Campus) and DSI HySA/Infrastructure, South Africa are gratefully acknowledged. This study forms part of the activities of the Unlohcked Consortium, which received funding from the Clean Hydrogen Partnership, Belgium under grant agreement No 101111964.

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