

# Hydrogen Generation via Catalytic Ammonia Borane Hydrolysis:

## A Patent Overview

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### Abstract

Hydrogen is one of the clean energies that will play an important role in energy selection for the coming low-carbon society. Ammonia borane ( $\text{NH}_3\text{BH}_3$ , abbreviated hereafter as AB) is considered as a promising hydrogen storage material due to its high hydrogen content (19.6 wt%), stability under ambient conditions (white powder), and favorable properties in dehydrogenation. AB can release hydrogen via pyrolysis, alcoholysis, hydrolysis, etc. Among these, AB hydrolysis is a safe and effective route, as it proceeds under mild conditions in the presence of an appropriate catalyst without the need for additional heat sources. For practical application, it is important to develop an efficient catalyst that further improves the hydrolytic dehydrogenation properties of AB. Many papers and reviews have been published, suggesting the research is at an advanced stage. On the other hand, since new technologies are often patented before they are put into practical use, the status of patent publication is of significant interest. This mini-review analyzes patent applications published up to 2023 on hydrogen generation via catalytic AB hydrolysis. The patent search yielded 99 patent publications, which were examined not only by country and research institution but also by two representative types of catalysts derived from noble metal (abbreviated hereafter as NM) and non-NM. Based on the analysis, future research and development was discussed.

Keywords: Catalysts, Ammonia Borane, Hydrolysis, Hydrogen, Patent Overview

## 1. Introduction

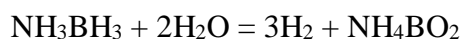
Fossil fuels are valuable resources that meet today's energy needs. However, in addition to being limited, they also pose problems such as air pollution, global warming, environmental destruction, and negative effects on human and animal health [1], [2], [3], [4], [5]. For these reasons, efforts are being accelerated to harness alternative cleaner energy (solar, wind, hydro, geothermal, nuclear, hydrogen, etc.) [6], [7], [8], [9], [10]. Solar and wind powers vary significantly depending on daily and seasonal conditions, resulting in intermittent energy supply. Hydro and geothermal powers are subject to strong geographical constraints. Concerns regarding nuclear power's long-term safety and environmental issues, including waste, have not been completely eliminated. In contrast, hydrogen can be stored and used continuously for power generation and combustion, to be an environmentally friendly zero-carbon energy resource that emits no greenhouse gases but only water when consumed. Currently, hydrogen is often produced from fossil fuels, however, in the near future, it is expected to be produced by the electrolysis of water using the aforementioned renewable energies. The idea of converting surplus electricity from renewable energy sources into hydrogen and storing it is gaining traction. If this is realized, so-called green hydrogen will become one of the main energy resources for a decarbonized society [11], [12], [13], [14], [15], [16], [17], [18].

Since hydrogen is a gas under ambient conditions, it faces difficult problems during storage and transportation [19], [20], [21], [22], [23]. Storing large amounts of gas in high-pressure tanks or as a liquid at extremely low temperatures requires corresponding special technology and large amounts of energy. Transporting by pipelines needs extensive infrastructure. Instead, there are two categories of other storage methods for hydrogen:

chemical storage and physical storage [24], [25], [26], [27], [28]. Chemical storage has the advantage of larger storage capacity than physical storage using adsorption, making it suitable for large-scale applications. It is a method of producing and transporting liquid or solid compounds from hydrogen, rather than hydrogen itself. Changing the shape to a liquid or solid reduces volume, making storage and transportation easier. It can accommodate a wide range of transportation methods, including land, sea, and air.

Known candidates for chemical storage include AB ( $\text{NH}_3\text{BH}_3$ ), ammonia ( $\text{NH}_3$ ), methylcyclohexane ( $\text{C}_6\text{H}_{11}\text{CH}_3$ ), formic acid ( $\text{HCO}_2\text{H}$ ), etc. [29], [30], [31], [32], [33], [34] AB can release hydrogen at room temperature when hydrolyzed with a metal catalyst. Due to its simple procedure and mild conditions, AB may be a favorable hydrogen storage material. AB is a solid substance (white powder) at ambient conditions and is characterized by high hydrogen density (19.6 wt%  $\text{H}_2$ ), low molecular weight (30.7 g/mol), storage safety, high solubility in water, and long-term stability in an aqueous solution [35], [36], [37], [38], [39]. The first synthesis of AB was reported in 1955, and several improved methods have been reported since then [40], [41], [42], [43], [44], [45], [46], [47], [48]. Besides hydrolysis, pyrolysis and alcoholysis can also dehydrogenate AB, however, hydrolysis has the advantages of a lower reaction temperature than pyrolysis and a faster hydrogen release rate than alcoholysis [49], [50], [51], [52], [53], [54], [55], [56], [57], [58].

The reaction equation for AB hydrolysis is as follows [59], [60], [61], [62]. AB is thought to be adsorbed on the catalyst surface, and H in AB combines with H in  $\text{H}_2\text{O}$  to form  $\text{H}_2$ , leaving non-polluting ammonium borate. One equivalent of AB can afford three equivalents of hydrogen. The two hydrogen atoms in the released hydrogen come from AB and  $\text{H}_2\text{O}$  molecules.



Hydrogen generation via catalytic AB hydrolysis can be an attractive approach to meet the needs of the environmentally friendly market with low costs and high hydrogen release rates. It is expected that the technology will be put to practical use as a hydrogen source for emergency power batteries and as an online hydrogen supply for hydrogen fuel cell vehicles.

Currently, typical catalysts for AB hydrolysis are derived from NM and non-NM, respectively, as described in the precedent reviews [63], [64], [65], [66], [67], [68], [69], [70], [71], [72], [73], [74], [75], [76], [77], [78], [79], [80], [81], [82], [83]. Although NM catalysts generally exhibit high catalytic activity, their high cost and rarity make them unsuitable for large-scale industrial applications. Instead, it seems meaningful to develop more cost-effective catalysts by using inexpensive transition metal elements. And, an additional useful strategy is to reduce the particle size of metal catalysts into nanoparticles (NPs) to improve their dispersibility and stability on various support materials, thereby increasing the catalytic activity while at the same time reducing the amount of metal catalysts used. As shown in the reviews, overall catalytic activity depends on metal NPs, the support, and their interactions. Therefore, research vectors can be roughly divided into two directions. One is research that uses highly active but expensive NM catalysts to pursue even higher activity while reducing the amount used. The other is research that realizes a high activity equivalent to NM catalysts using inexpensive non-NM catalysts. The development of efficient, cost-effective, and stable catalysts is a bottleneck in catalytic AB hydrolysis.

By the way, patents are often filed when new technologies are put into practical use, so it is important to know the publication status of patent applications. This mini-review searched for patents on hydrogen generation via catalytic AB hydrolysis published up to 2023 to extract 99 patents with experimental data on AB hydrolysis. Then, the status was investigated not only by country and research institution but also by two representative types

of catalysts, such as NM and non-NM catalysts. Based on the analysis, the future development of the research was envisioned from the viewpoint of practical application.

## 2. Materials and Methods

A patent search was performed using the free patent databases, PATENTSCOPE provided by the World Intellectual Property Organization (WIPO) and Google Patents presented by Google, mainly with the former for search and the latter for reference. It should be noted that the search covered all patent applications on catalytic AB hydrolysis published up to 2023, regardless of whether the patent has been granted. As is known, a patent has two dates. The filing date is the date on which a patent application was filed, and is an important indicator from the perspective of patentability, as those who file first can obtain the patent. While, the publication date is the date on which a patent application is published after a certain period of incubation, which is usually one and a half years after filing (the priority claim date). It is difficult to quantitatively discuss the recent situation based on the application year due to the incubation period. Hence, the discussion here is based on the publication year.

To minimize search omissions, the search was conducted twice using different combinations of four keywords: catalyst, AB, hydrolysis, and hydrogen. Patent families were set up so that only the representative could be hit. The first search aimed to find verbatim patents with the three keywords: catalyst, AB, and hydrolysis, which is a straightforward search to afford 93 hits. The second one was carried out using two keywords: AB and hydrogen, to search broadly in the related fields to yield 359 hits. Both hits were examined one by one to select patents containing experimental data on catalytic AB hydrolysis while removing duplicates. Patents that mentioned AB hydrolysis but did not include experimental data on it were screened out. Finally, 99 patents were selected for detailed analysis in the next section.

### 3. Results and Discussion

#### 3.1. Regional characteristics

The selected patents were divided by country based on the applicant. Also, they were classified into two categories: NM catalyst (blue) and non-NM catalyst (red) depending on the catalyst used. Fig. 1a shows that the patents were published in five countries, with China (CN) having the highest number of 90 (ratio of NM and non-NM catalysts = 45:45), followed by Japan (JP) with 3 (1:2), South Korea (KR) with 3 (2:1), the United States (US) with 2 (2:0), and Saudi Arabia (SA) with 1 (1:0). Fig. 1b is a pie chart showing the percentages, with China accounting for 91% of the total and the remaining four countries together accounting for 9%. Fig. 1a-b show that the patents are actively published in Asia, especially in China. It would be said that more countries are expected to participate to promote and diversify the research.

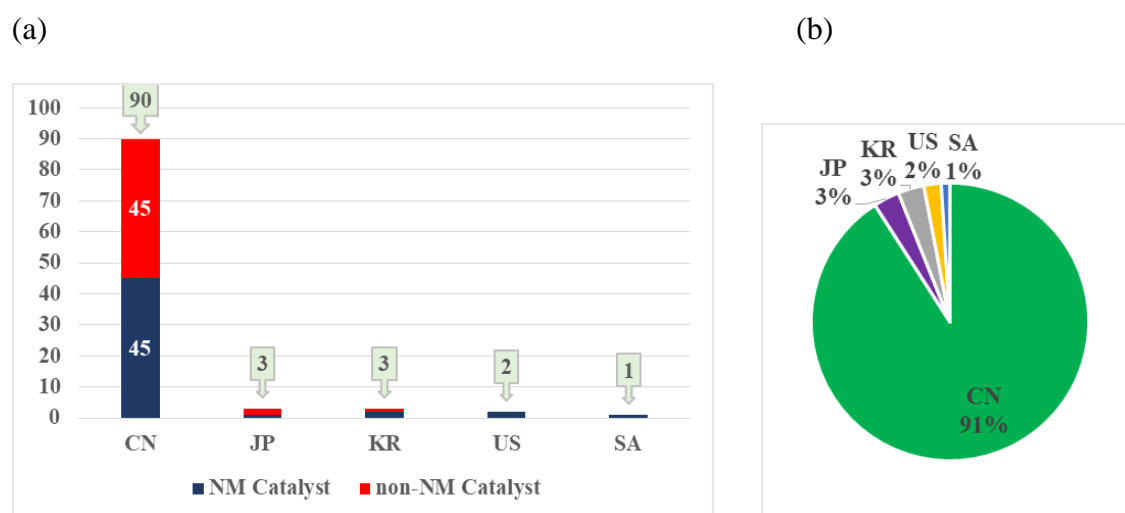


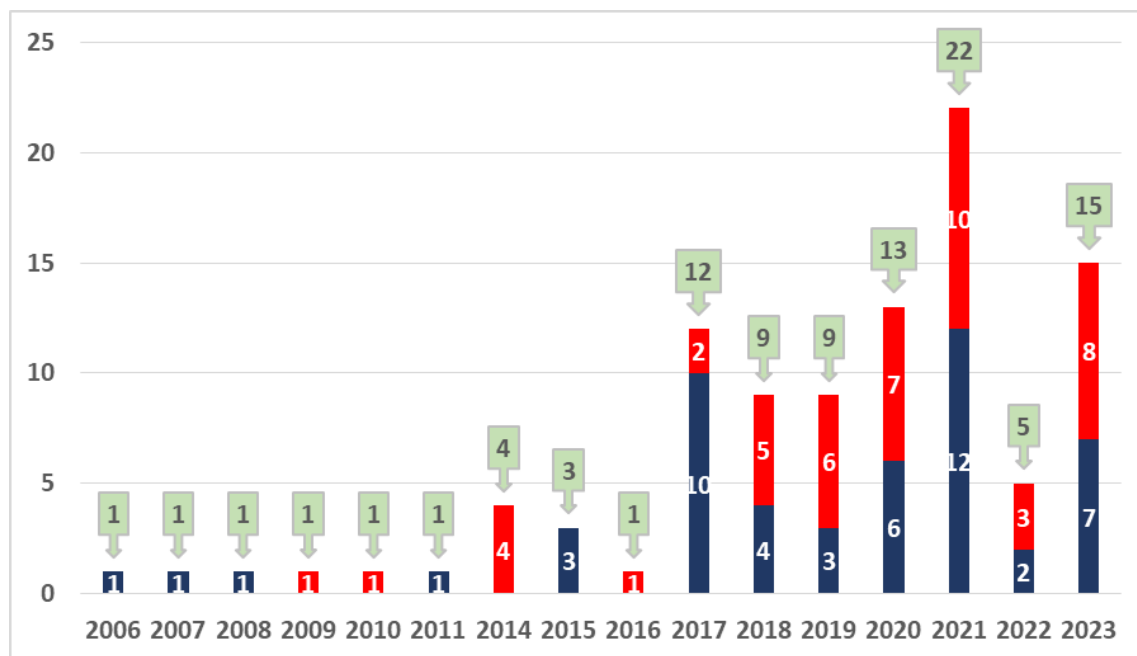
Fig. 1. Patent publication by country: (a) number; (b) percentage

#### 3.2. Annual patent publication and ratio of NM catalyst and non-NM catalyst

The annual trends of patent publication are shown in Fig. 2a, suggesting that the number seems to be increasing year by year, although it is not linear. For reference, the annual trends based on application year are also shown in Fig. 2b. Fig. 2a-b have similar shapes regardless of the year of publication or application. This is because the patents are published

early by the applicants without waiting for the incubation period to expire. Possible reasons include making it easier to find joint research partners, such as companies, and publishing the research results sooner.

(a)



(b)

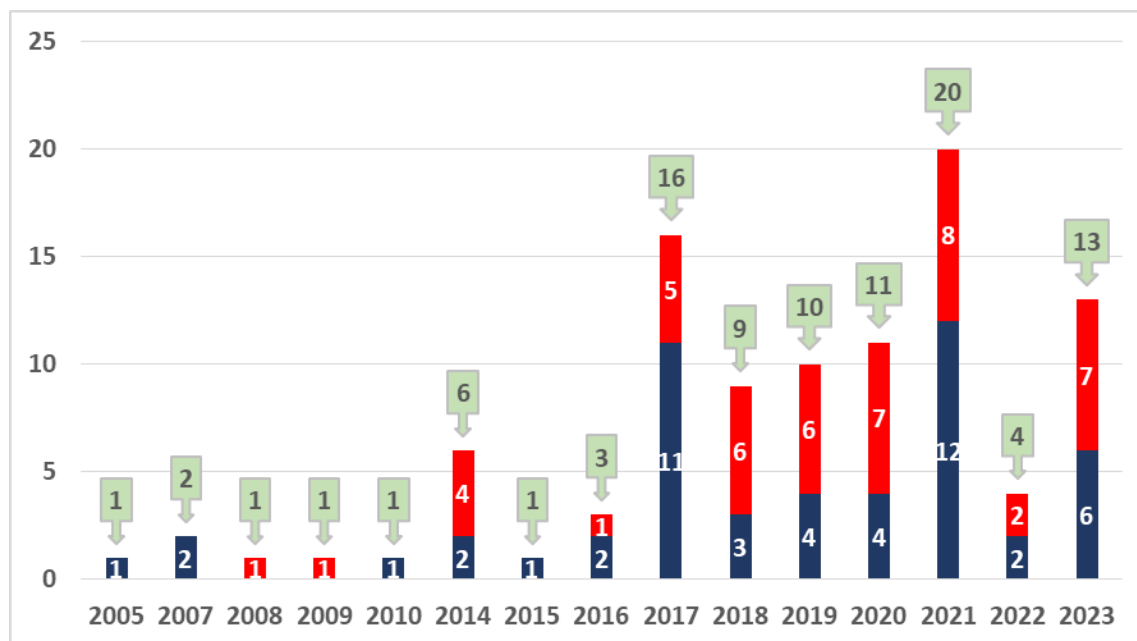


Fig. 2. Annual patent publication based on (a) publication year; (b) application year

The ratio of NM catalyst to non-NM catalyst patents was 51:48, as shown in Fig. 3a. In cases where the catalyst consists of both NM and non-NM, it is classified as an NM catalyst. To investigate any bias towards catalysts, the patents were divided into two parts: the first half (2006-2019, 44 patents) and the second half (2020-2023, 55 patents). As shown in Fig. 3b-c, the ratio of NM catalyst and non-NM catalyst was 24:20 in the first half and 27:28 in the second half. Comparing Fig. 3a-c, no major differences were found. Alternatively, the proportion of non-NM catalysts may increase slightly in the latter half. The true trend will become clear in the future. Currently, two types of catalysts are the main focus of the research.

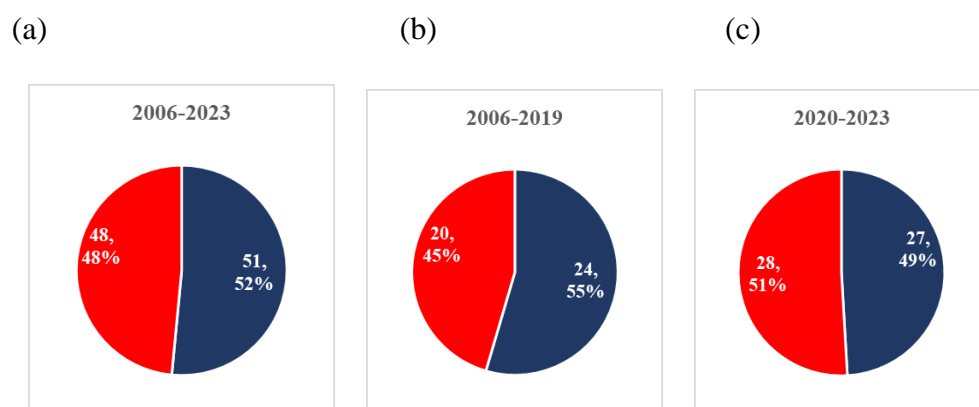


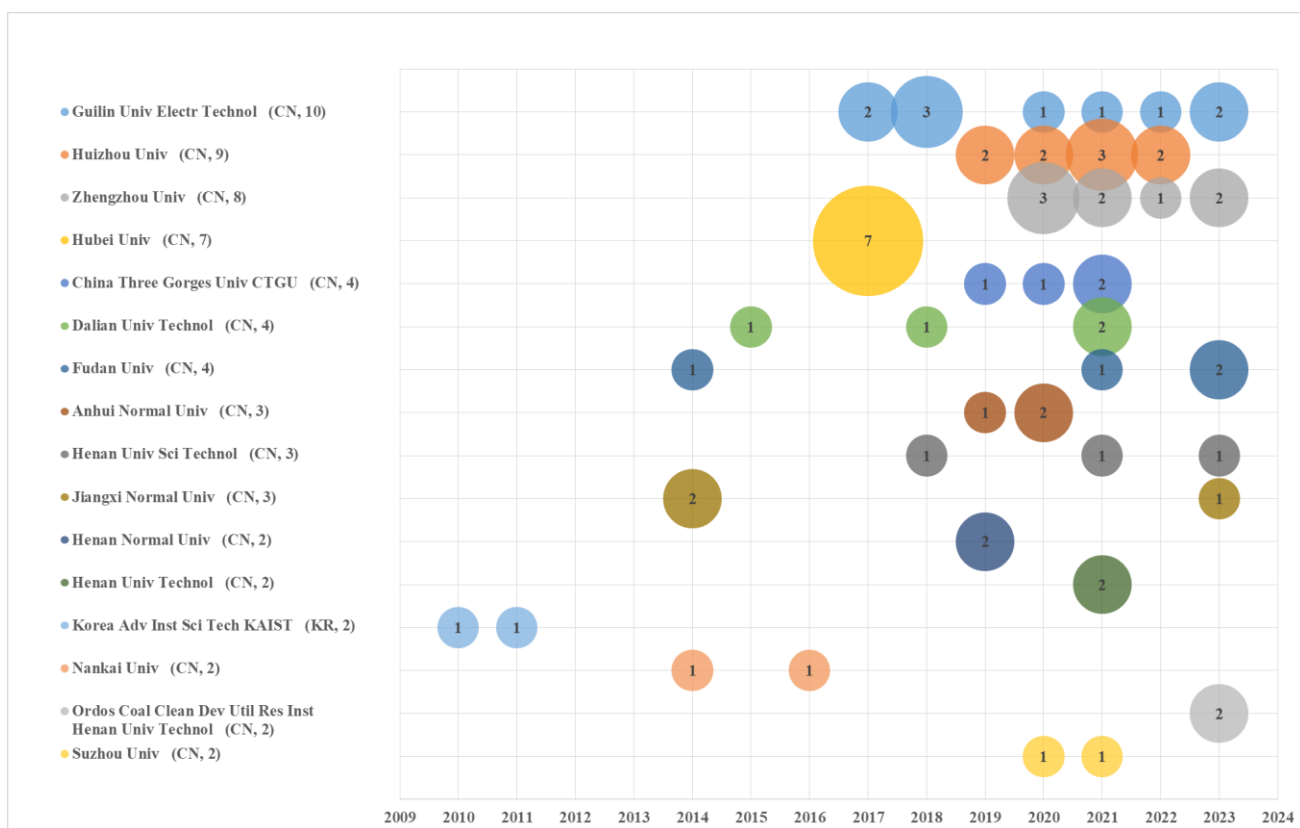
Fig. 3. Ratio of NM catalyst and non-NM catalyst in (a) 2006-2023; (b) 2006-2019; (c) 2020-2023

### 3.3. Applicants (research institutions)

16 applicants have published two or more papers, which are summarized in a bubble chart (Fig. 4a). It should be noted that the name of the applicant is at the time of submission, without taking into account subsequent changes. A larger bubble indicates a higher number of publications. All 16 applicants were from academia, of which 15 were from Chinese university institutions. The remaining one was a research institute in South Korea. The total publication number of the 16 applicants was 67, accounting for 68% of the total. Guilin Univ



(a)



(b)

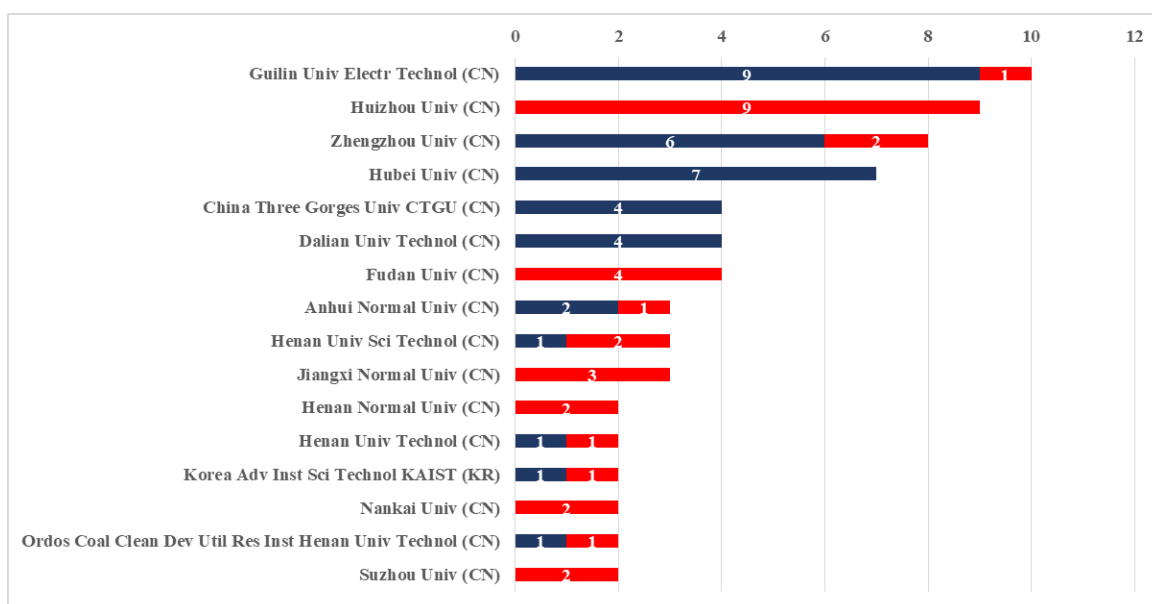


Fig. 4. Patent publication by 16 applicants: (a) annual number; (b) ratio of NM catalyst and non-NM catalyst

Electron Technol published the most patents with 10, followed by Huizhou Univ with 9 and Zhengzhou Univ with 8. As is clear from Fig. 4a, these top three applicants have been publishing patents on an annual basis in recent years, indicating that active research and patent application/publication are continuing. The next Hubei Univ published seven patents and is ranked fourth overall, but it published all of them in 2017 and has not published any since then. A similar phenomenon is observed at Korea Adv Inst Sci Technol KAIST and Nankai Univ. At these institutions, the related research might have been reduced or discontinued. For the 16 applicants, the publication ratio on NM catalyst and non-NM catalyst was investigated, as shown in Fig. 4b. Seven applicants published on both catalysts, while nine published on either one, suggesting that each catalyst is patentable.

### 3.4. Catalysts and properties

The properties of NM and non-NM catalysts described in the patent were summarized in Table 5a and 5b, respectively. The hydrogen generation activities of catalysts were expressed as Turnover Frequency (TOF:  $\text{min}^{-1}$ ), Temp (C), and Activation Energy ( $E_a$ ; KJ/mol). TOF is widely accepted as an indicator for catalyst evaluation ( $\text{H}_2 \text{ mol/catalyst mol/min} = \text{min}^{-1}$ ). Durability refers to activity after cycle use, expressed as Residual Activity (%/cycles).

As shown in Table 5a, in the mid-2000s, Xu et al. of Natl Inst Adv Ind Sci Technol AIST (JP) [84], Ramachandran et al. of Purdue Res Found (US) [85], and Mohajeri of Univ Cent Florida Res Found Inc (US) [86] disclosed that some NM (Pt, Pd, and Rh) and non-NM (Co and Ni) catalysts mediate AB hydrolysis to generate hydrogen. The reaction was completed in 3-360 min with a conversion rate of >90%. Since then, efforts have continued to improve the activity and durability of catalysts, especially by supporting them with carriers (Table 5a-b). By dispersing the metal catalyst as NPs on carriers, the specific surface area of

the catalyst increases, the interaction between the metal and the carrier is strengthened, and the deactivation due to possible aggregations is suppressed. The metal catalyst can be in the form of NPs, nanorods, nanowires, nanoclusters, nanospheres, microspheres, or the like. Carriers include metal-organic framework (MOF), carbon, graphene, carbon nanotubes (CNTs), carbon nitride (CN), molybdenum carbide (MoC), dendrimers, metal oxides (SiO<sub>2</sub>, CeO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>), boron nitride (h-BN), zeolite, carbon dots (CD), and composite atomic layer compounds (MXene), as well as their doped, oxidized/reduced, porous, and hollow materials. A detailed explanation of the functions of these carriers for AB hydrolysis has already been extensively discussed in notable reviews [63], [64], [65], [66].

In Table 5a, among the 30 disclosed TOFs achieved with NM catalysts, the maximum value is 4797.5, the minimum value is 37.951, and the average is 511 min<sup>-1</sup>. The four-digit TOFs of 4797.5, 1370, and 1327 min<sup>-1</sup> were recorded by Ru loaded on cubic CoCu spinel/carbon catalyst (Ru/Co<sub>2.28</sub>Cu<sub>0.72</sub>O<sub>4</sub>/C7.5) disclosed in 2023 by Jiang et al. of Chongqing Technol Bus Univ (CN) [132], Ru NPs dispersedly grown on TiO<sub>2</sub> with rich oxygen vacancies (Ru/TiO<sub>2-x</sub>) disclosed in 2020 by Li et al. of Zhengzhou Univ (CN) [111], and RuNi monoatomic alloy grown in situ on Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> carrier (RuNi<sub>SAA</sub>-Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub>) disclosed in 2023 by Liu et al. of Ordos Coal Clean Dev Util Res Inst Henan Univ Technol (CN) [131], respectively.

In Table 5b, among the 16 disclosed TOFs achieved with non-NM catalysts, the maximum value is 143.9, the minimum value is 8.4, and the average is 60 min<sup>-1</sup>. Over 100 TOFs of 143.9, 119.5, 112.3, and 111.06 min<sup>-1</sup> were displayed by Ni NPs deposited on MoO<sub>2</sub>-SBA-15 carrier (Ni/MoO<sub>2</sub>-SBA-15) disclosed in 2023 by Lu et al. of Jiangxi Normal Univ (CN) [178], Cu-Ni cobaltate nanowires (Cu<sub>0.6</sub>Ni<sub>0.4</sub>Co<sub>2</sub>O<sub>4</sub>) disclosed in 2019 by Li et al. of Huizhou Univ (CN) [151], Co doped ZIF-67 (zeolitic imidazolate framework) metal-organic framework (Co@ZIF-67) disclosed in 2021 by Liao et al. of Sichuan Univ (CN) [166], and

polyhedral Cu and P co-doped cobalt oxide nanocomposite (P-Cu-Co<sub>3</sub>O<sub>4</sub>@C) disclosed in 2021 by Yu et al. of Fudan Univ (CN) [170], respectively. Comparing both catalysts, the NM catalysts outperform the non-NM catalysts, which is consistent with previous results shown in the above reviews.

Table 5a. Summary of NM catalysts and properties

Applicant	Catalyst	Hydrogen Generation Activity			Durability	Year	Ref
		TOF (min <sup>-1</sup> )	Temp (C)	Ea (KJ/mol)	Residual Activity (%/cycles)		
Natl Inst Adv Ind Sci Technol AIST (JP)	PtO <sub>2</sub> Pt K <sub>2</sub> PtCl <sub>4</sub> PdSO <sub>4</sub> Pd NiSO <sub>4</sub> CoSO <sub>4</sub> [Rh(1,5-COD)(u-Cl)] <sub>2</sub>		RT		Pt : 99/2	2006	84
Purdue Res Found (US)	RuCl <sub>3</sub> .xH <sub>2</sub> O (0.05%) PdCl <sub>2</sub> (5%) CoCl <sub>2</sub> (5%)		RT			2007	85
Univ Central Florida Res Found Inc (US)	H <sub>2</sub> PtCl <sub>6</sub> Na <sub>2</sub> PtCl <sub>6</sub> K <sub>2</sub> PtCl <sub>6</sub>		25	83.9		2008	86
Korea Adv Inst Sci Technol KAIST (KR)	Ni <sub>0.72</sub> Pt <sub>0.28</sub> -NDCNTs		25	9.7		2011	87
East China Univ Sci Technol (CN)	SiW <sub>12</sub> O <sub>40</sub> <sup>5-</sup> -Pt/CNTs		30			2015	88
King Abdullah Univ Sci Technol KAUST (SA)	NiAgPd/C	93.8	21 (RT)	38.36	94/5	2015	89
Dalian Univ Technol (CN)	[Cp*Ir(6-Me-py-pz)(H <sub>2</sub> O)]SO <sub>4</sub>		65			2015	90
Hubei Univ (CN)	RuCuCo@MIL-101	241.2	25	48	54/5	2017	91
Hubei Univ (CN)	RuNi/TNTs	365.6	25	35.18	66.7/5	2017	92
Inst Adv Technol Univ Sci Technol China (CN)	Rh/VO <sub>2</sub>		60			2017	93
Hubei Univ (CN)	RuNi@MIL-110	454.7	25	36.0	76.0/5	2017	94
Hubei Univ (CN)	PdNi/TiO <sub>2</sub>	105.36	RT	24.86	64.0/5	2017	95
Guilin Univ Electron Technol (CN)	CoAg/nano-fiber				88.78/5	2017	96
Hubei Univ (CN)	RuCo@MIL-110	533.2	RT	31.7	79.0/5	2017	97
Hubei Univ (CN)	RuP@UIO-66	396.04	RT	33.15	54.6/5	2017	98
Hubei Univ (CN)	Ag <sub>0.1</sub> Co <sub>0.1-0.3</sub> @NH <sub>2</sub> -MIL-125		RT	26.79		2017	99
Lanzhou Univ (CN)	Pt/CCF	198.21	RT			2017	100

Dalian Univ Technol (CN)	$[(\eta^6\text{-C}_6\text{H}_6)\text{RuCl}_2(\text{bpym})][\text{Cl}]_2$	341.3	85			2018	101
Guilin Univ Electron Technol (CN)	Pt/ZIF-67	72.29	25	39.42		2018	102
Guilin Univ Electron Technol (CN)	Ru/C		25		99.1/4	2018	103
Guilin Univ Electron Technol (CN)	Ru/C		RT	23.86	58/5	2018	104
First Affiliated Hosp Zhengzhou Univ (CN)	RuCo@HCSs	180	25	19.11	81/7	2019	105
Jilin Univ (CN)	porous Rh nanospheres	47.3				2019	106
China Three Gorges Univ CTGU (CN)	Rh/graphene quantum dot					2019	107
Anhui Normal Univ (CN)	half-sandwich Ru & 2, 6-dithione substituted pyridine complex		25			2020	108
Guilin Univ Electron Technol (CN)	Ru-CA/CoNi-LDO*	463.9	RT	25.94		2020	109
China Three Gorges Univ CTGU (CN)	RhNP@THPP					2020	110
Zhengzhou Univ (CN)	Ru/TiO <sub>2-x</sub>	1370	25			2020	111
Anhui Normal Univ (CN)	PdCu/ZIF-67@ZIF-8	37.951	30	38.78	~100/5	2020	112
Zhengzhou Univ (CN)	CoRu <sub>0.5</sub> /CQDs		25	39.29		2020	113
Dalian Univ Technol (CN)	Pd/SiO <sub>2</sub>		25			2021	114
Zhengzhou Univ (CN)	PtNiO <sub>x</sub> /TiO <sub>2</sub> -VO		25			2021	115
Dalian Inst Chem Phys Chin Acad Sci (CN)	Pt/TiO <sub>2</sub>		25			2021	116
China Three Gorges Univ CTGU (CN)	Co-Ru/C*					2021	117
Zhengzhou Univ (CN)	Ru <sub>1-x</sub> Co <sub>x</sub> /P25	973.0	25			2021	118
Guizhou Univ (CN)	Rh/Al <sub>2</sub> O <sub>3</sub> -CTAB-400	107	RT			2021	119
Guilin Univ Electron Technol (CN)	Ru/Co-PCP	243.4	25	31.25	72.8/10	2021	120
Henan Univ Technol (CN)	alk-Ti <sub>3</sub> C <sub>2</sub> MXene-Pd	230.6	25			2021	121
Dalian Univ Technol (CN)	Ru <sub>50</sub> Fe <sub>12</sub> Co <sub>38</sub>	62	RT			2021	122
Nanjing Univ Sci Technol (CN)	PdCo@HCN	446.11	30	43.7		2021	123
China Three Gorges Univ CTGU (CN)	RhNP/TPP-PEG					2021	124
Henan Univ Sci Technol (CN)	Pt-Cu		RT			2021	125
Guilin Univ Electron Technol (CN)	Ru-Ni/rGO-PVP-1	422.7	25	21.1	82.1/20	2022	126
Ind Acad Cooperation Found Kyung Hee Univ (KR)	Pt <sub>90</sub> Pd <sub>90</sub> /dendrimer	108.5	25	47.8		2022	127
Henan Agric Univ (CN)	Pd <sub>0.1</sub> Cu <sub>0.9</sub> /T-PC	279	25		80/5 49/10	2023	128
Zhengzhou Univ (CN)	PdCoO <sub>x</sub> P25	210	25			2023	129

Guilin Univ Electron Technol (CN)	Ru/Co <sub>2</sub> P/N-CNT	193.9	25	32.4	42/5	2023	130
Ordos Coal Clean Dev Util Res Inst Henan Univ Technol (CN)	RuNi <sub>5</sub> AA-Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub>	1327	30			2023	131
Chongqing Technol Bus Univ (CN)	Ru/Co <sub>2.28</sub> Cu <sub>0.72</sub> O <sub>4</sub> /C <sub>7.5</sub>	4797.5				2023	132
Guilin Univ Electron Technol (CN)	Ru/CoP-NC	370.4	25	37.04	53/5	2023	133
Zhengzhou Univ (CN)	Ru/CNT@CN	643.6	25			2023	134

\* Magnetic property is noted in the patent.

Table 5b. Summary of non-NM catalysts and properties

Applicant	Catalyst	Hydrogen Production Activity			Durability	Year	Ref
		TOF (min <sup>-1</sup> )	Temp (C)	Ea (KJ/mol)	Residual Activity (%/cycles)		
Peking Univ (CN)	Ni-MOF		25		~100/20	2009	135
Korea Adv Inst Sci Technol KAIST (KR)	Co-P		30	21.6		2010	136
Nankai Univ (CN)	Ni <sub>3</sub> B/C		RT	44.06		2014	137
Fudan Univ (CN)	PEI-GO/Co	39.9	25	20.6		2014	138
Jiangxi Normal Univ (CN)	Cu@mSiO <sub>2</sub>		25			2014	139
Jiangxi Normal Univ (CN)	Ni-CeO <sub>2</sub> @graphene	53.6	RT			2014	140
Nankai Univ (CN)	CoN-C-700		25	31.0	97.2/10	2016	141
Guilin Univ Electron Technol (CN)	Co/C		25		97/4	2017	142
Fuzhou Univ (CN)	Ni@h-BN*					2017	143
Tech Inst Phys Chem Chin Acad Sci (CN)	Ni <sub>3</sub> FeN@SiO <sub>2</sub>	14.17				2018	144
Tianjin Univ (CN)	NiP		25			2018	145
Shenyang Normal Univ (CN)	Co-Mo-B		25	41.7	61.9/5	2018	146
Henan Univ Sci Technol (CN)	TiN*		25	38.76		2018	147
Shanxi Jiexiangrui Technol Co Ltd (CN)	Co/NC		25			2018	148
Anhui Normal Univ (CN)	Co-N-C@SiO <sub>2</sub> A	8.4	25	36.1		2019	149
Henan Normal Univ (CN)	nanometer Fe		RT			2019	150
Huizhou Univ (CN)	Cu <sub>0.6</sub> Ni <sub>0.4</sub> Co <sub>2</sub> O <sub>4</sub>	119.5	25			2019	151
Huizhou Univ (CN)	Co <sub>0.8</sub> Cu <sub>0.2</sub> MoO <sub>4</sub>		25			2019	152
Univ Hyogo (JP), Sanalloy Ind Co (JP)	Co-W-C					2019	153
Henan Normal Univ (CN)	monometallic nanometer Cu		RT			2019	154
Chengdu Univ Technol (CN)	Co/aza graphene	54	25	27.1		2020	155
Huizhou Univ (CN)	Cu <sub>0.6</sub> Co <sub>2.4</sub> (PO <sub>4</sub> ) <sub>2</sub>		25			2020	156
Hebei Univ Technol (CN)	Co-Cu*		25			2020	157
Huizhou Health Sci Polytech (CN) Huizhou Univ (CN)	Cu <sub>0.5</sub> Ni <sub>0.5</sub> Co <sub>2</sub> O <sub>4</sub>		25			2020	158

Zhengzhou Univ (CN)	Ni <sub>1.2</sub> Fe <sub>0.8</sub> @CN-G	25				2020	159
Suzhou Univ (CN)	Ni <sub>3</sub> Fe <sub>7</sub> P				>90/11	2020	160
Huizhou Univ (CN)	Cu <sub>2</sub> O-CoO	25				2020	161
Cent South Univ (CN)	Cu-Co oxide	25				2021	162
Huizhou Univ (CN)	Co <sub>3</sub> O <sub>4</sub> /CuMoO <sub>4</sub>	25				2021	163
Huizhou Univ (CN)	Cu cobaltate	25				2021	164
Suzhou Univ (CN)	R-Fe <sub>2</sub> O <sub>3</sub> Foam800				95/6	2021	165
Sichuan Univ (CN)	Co@ZIF-67*	112.3				2021	166
Henan Univ Technol (CN)	Ni-Mo <sub>2</sub> C/γ-Al <sub>2</sub> O <sub>3</sub>	80.9	30			2021	167
Changzhou Univ (CN)	Co <sub>3</sub> O <sub>4</sub> -CoCuO <sub>2</sub>	25				2021	168
Huizhou Univ (CN)	Co <sub>x</sub> Cu <sub>1-x</sub> Co <sub>2</sub> O <sub>4</sub> @Co <sub>y</sub> Cu <sub>1-y</sub> Co <sub>2</sub> O <sub>4</sub>	25				2021	169
Fudan Univ (CN)	P-Cu-Co <sub>3</sub> O <sub>4</sub> @C	111.06	30	38.31	68/5	2021	170
CQC Intime Testing Technol Co Ltd (CN)	Co/C		RT	52.02	70~80/50	2021	171
Huizhou Univ (CN)	Co/Sn*	23	RT			2022	172
Huizhou Univ (CN)	m-Co <sub>8</sub> CuNi@NC*					2022	173
Zhengzhou Univ (CN)	O-(CoP/Co <sub>2</sub> P)@SC	35	30			2022	174
Hyogo Public Univ Corp (JP), Sanalloy Ind Co Ltd (JP)	Co-W-C*		35			2023	175
Xiamen Univ (CN)	CuFeCo <sub>3</sub>	30.12	25	26.6	>90/10	2023	176
Yangzhou Polytech Inst (CN)	Cu-IL		40		97/5	2023	177
Jiangxi Normal Univ (CN)	Ni/MoO <sub>2</sub> -SBA-15	143.9	25			2023	178
Fudan Univ (CN)	Br-CoP-C	67.3	25	23.48		2023	179
Ordos Coal Clean Dev Util Res Inst Henan Univ Technol (CN)	CoP/h-BN <sub>OH</sub>	43	30			2023	180
Henan Univ Sci Technol (CN)	CoMoP-NC		25			2023	181
Fudan Univ (CN)	Co@MoO <sub>3</sub>	19.0	25			2023	182

\* Magnetic property is noted in the patent.

The reaction temperature is generally set at room temperature (RT) for both catalysts, which will be intended to emphasize that the catalytic AB hydrolysis proceeds under mild conditions and to suggest that no additional energy is required to initiate.

E<sub>a</sub> showed no significant difference between both catalysts. Among the 22 E<sub>a</sub>'s disclosed for NM catalysts, the maximum value is 83.9, the minimum value is 9.7, and the average is 37 KJ/mol. The three lowest E<sub>a</sub>'s are 9.7 [87], 19.11 [105], and 21.1 [126] KJ/mol. Meanwhile, among the 12 E<sub>a</sub>'s disclosed for non-NM catalysts, the maximum value is 52.02, the minimum value is 20.6, and the average is 33 KJ/mol. The three lowest E<sub>a</sub>'s are 20.6

[138], 21.6 [136], and 23.48 [179] KJ/mol. It should be noted that the lowest  $E_a$  of 9.7 KJ/mol was observed for the NM catalyst [87].

Durability is estimated in two ways in the patents: a quantitative evaluation incorporating time factors such as TOF, and a qualitative evaluation using a conversion rate as an index. The residual activities listed in Table 5a-b are the quantitative evaluation. Among them, the activity after five cycles is shown in 13 patents for NM catalysts and 3 patents for non-NM catalysts, respectively. The 13 data points for NM catalysts are 42 [130], 53 [133], 54 [91], 54.6 [98], 58 [104], 64.0 [95], 66.7 [92], 76.0 [94], 79.0 [97], 80 [128], 88.78 [96], 94 [89], and ~100 [112] %. The 3 data points for non-NM catalysts are 61.9 [146], 68 [170], and 97 [177] %. Comparing them, NM catalysts might be more easily deactivated than non-NM catalysts. Catalyst deterioration is generally unavoidable. Higher repeatability is better, but it may be possible to adjust by using a large amount of catalyst in advance or by adding additional catalysts, while cost is an issue.

Regarding catalyst recovery, some cobalt- and nickel-derived catalysts are shown to be magnetic. There is a concern that it may result in agglomeration, however, it is an advantage that the catalyst can be recovered with it. As shown in Table 5a-b, nine catalysts are described as magnetic, while seven of them are non-NM catalysts [143], [147], [157], [166], [172], [173], [175]. The remaining two are the catalysts that contain both NM and non-NM, being classified as NM catalysts [109], [117]. Catalyst recovery using magnetic properties may be a useful means from the viewpoint of catalyst recycling.

#### 4. Conclusion

Hydrogen is gaining global acceptance as the next-generation energy vector, and solutions are being actively sought to overcome limitations such as storage and transportation. While chemical storage is a promising option, hydrogen carrier molecules with abundant



capacity are entering the commercial phase. AB is one such hydrogen carrier. Hydrogen generation via catalytic AB hydrolysis has attracted attention because it proceeds quantitatively under ambient conditions. Many research papers and reviews of them are published every year, as shown in the previous section. This mini-review investigated patent publication status through 2023. A patent search yielded 99 patents that contain experimental data on AB hydrolysis.

The status of applicants (research institutions) by country revealed that there are five countries in which the related patents were published (Fig. 1). China has the highest number with 90 (91%), followed by Japan with 3 (3%), South Korea with 3 (3%), the United States with 2 (2%), and Saudi Arabia with 1 (1%), indicating popular publications in Asia. It would be desirable for more countries to participate in order to promote research development and diversity.

Two types of catalysts are mainly utilized for AB hydrolysis: NM catalysts, which are highly active but expensive, and non-NM catalysts, which are relatively low in activity but inexpensive, and both are often dispersed in carriers. The total ratio of the two catalysts was 51 : 48. No significant difference in the ratio was observed between periods of the first half (2006-2019) and the second half (2020-2023). Patents on non-NM catalysts universally point out that expensive NM catalysts are unsuitable for practical use. If this were true, it would seem that the proportion of patents on non-NM catalysts would be higher, but in reality, they were almost equal.

All 16 institutions that have published two or more patents are from academia; 15 are Chinese university institutions, and the remaining one is a Korean institution, none of which are companies. The experimental scale was usually quite small, with tens of milligrams of catalyst used and hundreds of milliliters of hydrogen generated. These facts may mean that the research field is still at a basic stage. To demonstrate the utility of catalytic AB hydrolysis,

it would be desirable to scale up the experiments. Although joint development remains an option for academic institutions, companies are often responsible for social implementation. It is hoped that more companies will participate in applied research and patenting.

The research field may need a breakthrough, such as the discovery of a highly efficient catalyst that exceeds previous achievements or the creation of a scaled-up prototype product, leaving aside competitive advantages in the market. Even the first patent published in 2006 confirmed through experimental examples that the hydrogen generated could run a fuel cell [84]. Demonstration of not only catalyst recycling but also AB recycling will be necessary. These efforts could catalyze an increase in the number of participating institutions around the world and create a virtuous upward loop toward practical application.

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## **Conflicts of interest**

The author declares no conflict of interest.

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