Transformation of Glucose to 5-Hydroxymethylfurfural Over Regenerated Cellulose Supported Nb₂O₅·nH₂O in Aqueous Solution

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Abstract

Niobic acid $(Nb_2O_5 \cdot nH_2O)$ was immobilized on regenerated cellulose (Re-Cellulose) to afford a heterogeneous catalyst, termed $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose, that was characterized by powder X-ray diffraction, microscopy (SEM and TEM) and spectroscopic (Raman and FTIR) techniques. The surface acidity of the catalyst was determined using FTIR spectroscopy employing pyridine as a molecular probe. The $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst was investigated in the conversion of glucose into 5-hydroxymethylfurfural (HMF) in aqueous media. The catalyst can be reused several times without undergoing a significant loss in activity.

Graphic Abstract



Keywords Sustainable chemistry \cdot Heterogeneous catalysis \cdot Regenerated cellulose \cdot Niobium pentoxide \cdot Glucose \cdot 5-Hydroxymethylfurfural (HMF)

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1 Introduction

Humanity is confronted with the diminishing resources and increasing waste and pollution that is devastating the environment [1]. Consequently, many approaches are being considered to reduce the burden on the environment including the transformation of biomass into platform chemicals, which would reduce society's dependence on non-renewable petrochemical feedstocks [2, 3]. The conversion of various



biomass derivatives into 5-hydroxymethylfurfural (HMF) is of particular interest as HMF can be applied in the manufacture of polyurethanes analogous, polyamides and furanic polyesters [4–9]. Currently, the dehydration of small sugars such as glucose or fructose is the main route for the production of HMF. Both the catalyst and solvent play important roles in the reaction.

A large number of heterogeneous (Lewis acidic) catalysts such as mesoporous tantalum oxide [10], zeolites such as Fe/HY zeolite [11], ZSM-5 zeolites [12], and SAPO-34 [13] have been evaluated as catalysts for the conversion of glucose into HMF. Metal–organic framework (MOF) based catalysts such as MIL-101Cr [14], and other supported catalysts [15–18] have been also evaluated. In addition, different solvents including DMSO [13], THF [14] and ionic liquids [19, 20] have been used. Among the solvents used, water would be ideal from both cost and environmental perspectives, but tends to results in the lowest yields of HMF as most Lewis acidic catalysts are not water-tolerant [19]. Therefore, a water-tolerant heterogeneous catalyst for high HMF yield from glucose in water would meet an unmet need in the field.

Niobic acid (Nb₂O₅·nH₂O) is a water-tolerant metal-oxide [21], but when employed as a catalyst for the production of HMF from glucose in water, the yield of HMF is very low even when high catalyst loadings are employed (e.g. 12.1% yield of HMF from 10 mg/mL glucose in water at 393 K for 3 h at a Nb₂O₅ \cdot nH₂O loading of 100 mg/mL) [21]. Herein, we describe the synthesis of a catalyst comprising Nb₂O₅·nH₂O immobilized on regenerated cellulose (Re-Cellulose). Re-Cellulose was chosen as the support material because it is both abundant and can be easily modified [22-27]. Dissolution of cellulose in aqueous NaOH/urea solution at low temperature breaks the hydrogen bonding networks in cellulose. By adding the niobium catalyst to this solution, then regenerating the cellulose, Nb₂O₅·nH₂O becomes dispersed within the regenerated hydrogen bonding networks of the Re-Cellulose support (Fig. 1). The resulting catalyst shows good activity in the conversion of glucose to HMF.

2 Experimental

2.1 Materials and Methods

All chemicals were obtained from commercial sources and are of analytical grade and used as received. X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared (FTIR), Pyridine Fourier-transform infrared (Py-FTIR) and Scanning Electron Microscopy (SEM) were used to characterize the catalyst as described previously [15, 16]. Transmission Electron Microscopy



Fig. 1 Illustration of $Nb_2O_5 \cdot nH_2O$ catalyst locked in the regenerated hydrogen bond network in the Re-Cellulose support material

(TEM) was performed on a FEI Tecnai G2 f20 s-twin operating at 200 kV. Raman spectra were obtained using a French Horiba Jobin Yvon LabRAM HR800 Microconfocal Raman Spectrometer equipped with a thermoelectrically cooled charge coupled device (CCD) detector. The Nb content in the liquid phase was determined on an Agilent 7900 inductively coupled plasma-mass spectrometry (ICP-MS). Highperformance Liquid Chromatography (HPLC) analysis was performed on an Agilent 1200.

2.2 Preparation of Nb₂O₅·nH₂O@Re-Cellulose catalyst, Unsupported Nb₂O₅·nH₂O and Re-Cellulose.

2.2.1 Synthesis of the Nb₂O₅·nH₂O@Re-Cellulose Catalyst

The Nb₂O₅·nH₂O@Re-Cellulose catalyst was prepared using a three-step procedure:

- Step 1 Stock solution A containing cellulose (0.83 g), NaOH (2.96 g, 74.0 mmol) and urea (1.73 g, 28.8 mmol) in water (20 mL) was prepared according to a literature method [27] and has a cellulose concentration of 4.0% (w/w).
- Step 2 Stock solution B was prepared by addition of NbCl₅ (1.00 g, 3.7 mmol) into a mixture of 0.5 M HCl (30 mL) and ethanol (10 mL) under stirring at room temperature and then treated with ultrasound for 30 min [28].
- Step 3 Preparation of the Nb₂O₅·nH₂O@Re-Cellulose catalyst: Solution A (5 mL) and a solution B (5 mL) were mixed together and the resulting reaction mixture was irradiated with ultrasound for 10 min at room temperature. The cellulose was regenerated

(Re-Cellulose) from the mixture using a literature method [27], i.e. the mixture was placed in a hydrothermal reactor and heated at 453 K for 8 h. The resulting solid material was removed by filtration, washed with deionized water (4×15 mL) and dried under vacuum at 353 K to afford a powder (200 mg, 71% yield).

2.2.2 Synthesis of Unsupported Nb₂O₅·nH₂O

Solution B (5 mL) was placed in a hydrothermal reactor and heated at 453 K for 8 h. Then, the mixture was filtered, the solid washed with deionized water (4 × 15 mL), dried under vacuum at 353 K, to afford Nb₂O₅·nH₂O (68 mg, 83% yield).

2.2.3 Synthesis of Re-Cellulose

Solution A (5 mL) was heated in a hydrothermal reactor at 453 K for 8 h. The resulting precipitate was collected by filtration and washed with deionized water (4×15 mL), dried under vacuum at 353 K, to afford Re-Cellulose (190 mg, 95% yield).

2.3 Typical Procedure for the Catalytic Conversion of Glucose into HMF

In a typical reaction, an aqueous glucose solution (5%; w/w; 2.5 mL) and the desired amount of the Nb₂O₅·nH₂O@Re-Cellulose catalyst (80–240 mg, 3.2–9.6%, w/w) was added to a 25 mL sealed thick-walled glass reactor. The reaction mixture was heated at the required temperatures (413 K, 423 K or 433 K) for a given time (30 min–6.5 h). Next, the reaction was quenched by placing the reactor in a water bath at room temperature. The sample was filtered and then the filtrate analyzed using HPLC. For the recycling studies, the used Nb₂O₅·nH₂O@Re-Cellulose catalyst was separated from the reaction mixture by filtration, washed with deionized water (4×15 mL), dried under vacuum oven at 343 K for 8 h, and then reused.

2.4 Determination of the Reaction Products

The conversion of glucose and the yield of HMF were determined according to literature methods (also see the Supplementary Material) [15, 16].

3 Results and Discussion

3.1 Catalysts Characterization

The $Nb_2O_5 \cdot nH_2O@Re$ -Cellulose catalyst, the Re-Cellulose support and the unsupported $Nb_2O_5 \cdot nH_2O$ catalyst were

characterized by XRD (Fig. 2). The three materials display broad peaks, indicating that they are all amorphous, with the characteristics of Re-Cellulose and $Nb_2O_5 \cdot nH_2O$ present in the $Nb_2O_5 \cdot nH_2O$ @Re-Cellulose catalyst.

SEM of the Nb₂O₅·nH₂O@Re-Cellulose catalyst shows a loose and non-uniform surface (Fig. 3a) [29]. Element mapping confirms the presence of the C, O and Nb elements (Fig. 3b–d). Notably, niobium is uniformly distributed over the Re-Cellulose support (Fig. 3d.).

The TEM image of the Nb₂O₅·nH₂O@Re-Cellulose catalyst shows that the Nb₂O₅·nH₂O is encapsulated in the Re-Cellulose (Fig. 4a, see Fig. 4b for comparison with the Re-Cellulose support. The size of the Nb₂O₅·nH₂O@Re-Cellulose particles range from ca. 100 to 200 nm, larger than those usually reported [28, 30].

Further confirmation for the presence of Nb₂O₅·nH₂O in the catalyst was provided by the characteristic bands between 837 and 935 cm^{-1} in the Raman spectrum (Fig. 5) [31]. A band centered at ca. 678 cm⁻¹ is also observed, which can be assigned to bulk Nb₂O₅·nH₂O [32]. It has been suggested that the Nb₂O₅·nH₂O species are connected with supports containing hydroxyl groups (OH) through hydrogen bonding [33], and such interactions are observed in FTIR spectrum of the Nb₂O₅·nH₂O@Re-Cellulose catalyst, evidenced by the strong absorptions between 2800 and 3800 cm^{-1} (Fig. S1). It would appear that OH groups in both the Nb₂O₅·nH₂O and Re-Cellulose components are involved in the formation of hydrogen bonding networks (Nb-O-H···O(H)-cellulose or Nb–O(H)···HO-cellulose) [27]. In addition, the FTIR spectra of the catalyst and cellulose before and after regeneration (cf. Figs. S1 and S2) are similar, indicating that only minor changes to the hydrogen bonding network have taken place, which is as expected as the regenerated cellulose is formed



Fig. 2 The XRD spectra of the Nb_2O_5 $\cdot nH_2O@$ Re-Cellulose, Re-Cellulose and Nb_2O_5 $\cdot nH_2O$



Fig. 3 SEM (a) and element mapping (b-d) of the Nb₂O₅·nH₂O@Re-Cellulose catalyst



Fig. 4 The TEM images of the $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst (a) and the Re-Cellulose support (b)

only through a rearrangement of the hydrogen bonds [34, 35].

The FTIR spectra of pyridine adsorption and desorption on the $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst are shown in Fig. 6. The presence of Lewis acid sites in the

 $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst is evidenced by the peaks around 1445 cm⁻¹ in the desorption spectrum at 423 K and their concentration was estimated as 23.52 µmol/g. Brønsted acid sites do not appear to be present, due to the absence of a peak at around 1538 cm⁻¹ in the desorption



Fig. 5 The Raman spectra of the $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst and Re-Cellulose



Fig. 6 FTIR spectrum of pyridine adsorption and desorption on the Nb_2O_5 -nH₂O@Re-Cellulose catalyst

Table 1Conversion of glucose(and fructose) to HMF

spectrum. For comparison, the FTIR spectra of pyridine
adsorption and desorption on Re-Cellulose and Nb ₂ O ₅ •nH ₂ O
are shown in Figs. S3 and S4, respectively. No Lewis acid
sites in Re-Cellulose can be found (Fig. S3), whereas both
Brønsted acid sites and Lewis acid sites are present in
$Nb_2O_5 \bullet nH_2O$ (Fig. S4).

3.2 Catalytic Conversion of Glucose to HMF

The formation of HMF on Nb₂O₅•nH₂O does not proceed on Brønsted acid sites, but on Lewis acid sites [21]. Therefore, both the isomerization of glucose and the dehydration of fructose should proceed on the Lewis acid sites in the Nb₂O₅·nH₂O@Re-Cellulose catalyst. The Nb₂O₅·nH₂O@ Re-Cellulose catalyst was evaluated in the conversion of glucose to HMF in water, see Table 1. In the absence on the catalyst HMF was not observed (Table 1, entry 1) and in the absence of a substrate the Re-Cellulose support remained inert and did not afford HMF-note that some catalysts can transform cellulose into HMF [36]. At a catalyst loading of 8% (w/w) at 423 K, the conversion reached 98% and HMF was obtained in 27.8% yield after 4 h (Table 1, entry 2), whereas the unsupported Nb₂O₅ \cdot nH₂O catalyst results in a yield of only 7.2% of HMF under the same conditions (Table 1, entry 3). The enhanced activity of the Nb₂O₅·nH₂O@Re-Cellulose catalyst compared to Nb₂O₅·nH₂O may be due to the highly-dispersed nature of Nb₂O₅·nH₂O nanoparticles within the Re-Cellulose matrix. Note that the Re-Cellulose support material is catalytically inactive (Table 1, entry 4) and a much lower yield of HMF (6.7%) is obtained when a mechanical mixture of Re-Cellulose support (178 mg) with unsupported Nb₂O₅·nH₂O (22 mg) is used as the catalyst (Table 1, entry 5). The Nb₂O₅·nH₂O@Re-Cellulose catalyst was also evaluated in the conversion of fructose, with HMF obtained in 31.9% yield (Table 1, entry 6), demonstrating that the catalyst can be used to dehydrate other substrates. Moreover, the absence of Brønsted acid sites in Nb₂O₅·nH₂O@Re-Cellulose

No.	Catalyst	Substrate	Conversion (%)	HMF (%)
1	Blank	Glucose	_	_
2 ^a	$Nb_2O_5 \cdot nH_2O@Re-Cellulose$	Glucose	98.0	27.8
3 ^b	Unsupported Nb ₂ O ₅ ·nH ₂ O	Glucose	42.0	7.2
4 ^c	Re-Cellulose support	Glucose	-	-
5 ^d	Re-Cellulose and Nb ₂ O ₅ ·nH ₂ O	Glucose	36.7	6.7
6 ^e	$Nb_2O_5 \cdot nH_2O@Re-Cellulose$	Fructose	99.0	31.9

Reaction conditions: Aqueous solution of glucose (5 wt%; 2.5 mL), 423 K, 240 min, 500 rpm; ^a200 mg catalyst; ^b22 mg catalyst; ^c178 mg catalyst; ^dRe-Cellulose (178 mg) + the unsupported Nb₂O₅·nH₂O (22 mg). The content of the Nb₂O₅·nH₂O and the Re-Cellulose in the Nb₂O₅·nH₂O@Re-Cellulose was calculated according the atomic ratio determined via Energy Dispersive X-Ray Spectroscopy (EDS) (Table S1) and the Nb₂O₅·nH₂O@Re-Cellulose consists of 11% Nb₂O₅ mass ratio. ^eFructose solution (5 wt%; 2.5 mL), 423 K, 240 min, 500 rpm, 200 mg catalyst. Yields averaged from three experiments

contributes to the stability of HMF in water, which is also beneficial for the high yield of HMF.

3.3 Influence of the Reaction Temperature and Time

The influence of reaction temperature and time on the conversion of glucose and the yield of HMF and was studied, with the conversion of glucose reaching a maximum within 2 h at temperatures above 423 K (Fig. 7a). At 413 K, the conversion of glucose reaches a maximum after more than 4 h (Fig. 7a), and the yield of HMF is only 10.9% (Fig. 7b). The yield of HMF increases as the reaction temperature is increased with an optimal reaction temperature of 423 K (Fig. 7b). Beyond this temperature the yield of HMF decreases, as observed previously [15, 16].

3.4 Influence of the Catalyst Loading

Increasing the catalyst loading from 80 to 200 mg (3.2–9.6%, w/w) leads to an increase in the yield of HMF from 7.0 to 27.8% (Fig. 8). Further increasing of the catalyst loading to 240 mg (9.6%, w/w) leads to a decrease in the yield (24.3%). The optimal loading of the Nb₂O₅·nH₂O@Re-Cellulose catalyst is 8% (w/w). In accordance with other reports [16], a low catalyst loading does not provide sufficient active centers whereas too much catalyst leads to side reactions.

3.5 Recycling the Nb₂O₅⋅nH₂O@Re-Cellulose Catalyst

Due to the heterogeneous nature of the $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst in water, the catalyst can be easily recovered from the reaction mixture via filtration. After



Fig.8 Influence of the $Nb_2O_5{\cdot}nH_2O@$ Re-Cellulose catalyst loading on the yield of HMF

washing with deionized water, the recovered catalyst may be reused. The XRD pattern and Raman spectrum of the recovered Nb₂O₅·nH₂O@Re-Cellulose catalyst are almost identical to the freshly prepared catalyst (cf. Figs. S5 and S6). The recovered Nb₂O₅·nH₂O@Re-Cellulose catalyst was also examined by SEM which reveals an overall similarity with the freshly prepared catalyst (Fig. S7A) and element mapping confirms that the niobium remains highly dispersed (Fig. S7B).

Indeed, the high stability and facile separation of the $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst enables its successful recycling. After 4 catalytic runs, the conversion of glucose and the yield of HMF decreased only slightly from 98 to 93.5% and from 27.8 to 22.2%, respectively (Fig. 9).



Fig. 7 Influence of reaction temperature on the conversion of glucose and formation of HMF as a function of time employing the $Nb_2O_5 \cdot nH_2O@$ Re-Cellulose catalyst



Fig.9 Recycling of the $Nb_2O_5{\cdot}nH_2O@Re-Cellulose$ catalyst in the conversion of glucose to HMF

The slight deactivation of the Nb₂O₅·nH₂O@Re-Cellulose catalyst is possibly due to the formation of humins byproducts that adhere to the surface of the catalyst [16]. Importantly, ICP analysis of the filtrate after 4 catalytic runs showed < 5 ppm of niobium in the filtrate, confirming the stability of the catalyst.

3.6 Comparison of the Performance of the Nb₂O₅·nH₂O@Re-Cellulose Catalyst with Other Catalysts

The performance of the Nb₂O₅ \cdot nH₂O@Re-Cellulose catalyst in the conversion of glucose to HMF is compared with reported catalysts in Table 2. The yield of HMF (27.8%)

obtained with the Nb₂O₅·nH₂O@Re-Cellulose catalyst is considerably higher than that obtained with Sn-Beta/HCl (7.92%) [37] and SO₄/ZrO₂ (10.0%) [38] in water, and higher than other catalysts that operate in aqueous-organic mixtures (Table 2) [39–41]. Presumably the absence of Brønsted acid sites in the Nb₂O₅·nH₂O@Re-Cellulose catalyst contributes to the stability of HMF in water, which impacts strongly on the yield obtained.

4 Conclusion

A heterogeneous Nb₂O₅·nH₂O@Re-Cellulose catalyst was prepared and applied in catalytic conversion of glucose to HMF employing water as the solvent. The highly dispersed $Nb_2O_5 \cdot nH_2O$ nanoparticles in the catalyst, that contains Lewis acidic sites and not Brønsted acidic sites, is highly active for the conversion of glucose and fructose into HMF. The strong interactions between the Nb₂O₅ \cdot nH₂O nanoparticles and Re-Cellulose support involves extensive hydrogen bonding networks leading to high stability and limited leaching of the Nb₂O₅ \cdot nH₂O nanoparticles. The strategy described here to construction supported catalysts via the regeneration of cellulose could be extended to other metal oxide-based catalyst. In addition, it is likely that these catalysts can also be applied in other reactions that would benefit from being conducted in aqueous environments.

Table 2 Comparison of the
Nb ₂ O ₅ ·nH ₂ O@Re-Cellulose
catalysts with other catalysts
that operate in water, aqueous-
organic solutions or ionic
liquids for the conversion of
glucose to HMF

Catalyst	Solvent	Glucose con- centration (%) ^a	Tempera- ture (K)	Time (min)	Yield (%)	References
Nb ₂ O ₅ ·nH ₂ O@ Re-Cellulose	Water	5.0	423	240	27.8	This work
Sn-Beta/HCl	Water	10.0	413	120	7.9	[37]
SO ₄ /ZrO ₂	Water	0.5	373	360	10.0	[38]
FePO ₄	Water/THF	2.5	413	60	22.6	[39]
Beta-Cal750 ^b	Water/DMSO	2.4	453	60	20.2	[40]
TaOPO ₄ ^c	Water/MIBK ^c	3.0	443	60	18.5	[41]
FeCl ₃	[EMIM]Cl	6.0	353	180	10.0	[19]

^aGlucose Concentration = $m_{glucose}/(m_{glucose} + m_{solvent})$, $m_{glucose}$ is the quality of glucose and $m_{solvent}$ is the quality of solvent

^bBeta-Cal750: Beta-NH₄ is calcined in air at 1023 K

^cTaOPO₄ = mesoporous tantalum phosphate; MIBK = methyl isobutyl ketone)

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