Catalysis Communications 154 (2021) 106305

Contents lists available at ScienceDirect

Catalysis Communications

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

Short communication

A novel selective oxidative cleavage of C—C bond mediated by black nickel oxide in the presence of molecular oxygen

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ARTICLE INFO

Keywords:
Heterogeneous catalysis
1, 2-diols
C—C bond cleavage
Nickel oxide
Molecular oxygen

ABSTRACT

A selective aerobic oxidative cleavage of C—C bond is developed with black nickel oxide (NiO_x) as the catalyst. For the oxidation of 1-phenyl-1, 2-ethanediol, a 97.5% conversion in 96.7% selectivity of benzaldehyde is obtained under 0.3 MPa of O_2 at 140 °C for 3 h. The relationship between the catalytic performance of NiO_x and structure is discussed. It is concluded that the existence of Ni^{3+} should be crucial to the activity of catalyst. Moreover, the recycling experiments showed that the catalyst can retain a high activity even after being reused for five times.

1. Introduction

The selective oxidative carbon-carbon (C—C) bond cleavage of organic compounds is a very significant technology for converting large molecules to the smaller ones [1,2]. Further, this process is very useful for biomass transformation, considering that the existence of numerous C - C inter-unit linkage in the structure of cellulose, hemicellulose and lignin [3]. Generally, the oxidative cleavage reaction is considered to be a synthetic method to generate carbonyl groups in organic chemistry. In traditional methods, the inorganic oxidants such as potassium permanganate, lead tetraacetate, chromium trioxide, manganese dioxide, DMSO, sodium and potassium periodate were employed for oxidative cleavage processes under heating conditions [4–7]. For example, Sudalai's group found that the carbon-carbon bond cleavage of 1, 2-diols was achieved using an excess of $NaIO_4$ reagents in dichloromethane [5]. However, the use of these excess oxidants is disadvantageous and not popular in the present chemistry, owing to their high toxicity, difficult storage and high cost; meanwhile, the produced hazardous wastes were harmful to the environment. Therefore, it is necessary to develop a highly efficient, environment-friendly and economic approach, in which molecular oxygen as oxidant is clean and green for the oxidative processes.

In recent years, the oxidative cleavage of 1, 2-diols with molecular oxygen has been investigated. Therein, Wang et al. employed Pd $(OAc)_2$ and Na_2CO_3 as the catalyst to promote the carbon-carbon bond cleavage of 1, 2-diols where a 95% yield of benzaldehyde was acquired under the optimized conditions [8]. Subsequently, Neumann's group [9] found that the carbon-carbon bond cleavage of D-glucose was performed successfully with $H_5[PV_2Mo_{10}O_{40}]$ as the catalyst. Then, the aerobic efficient carbon-carbon bond cleavage of 1, 2-diols was achieved using vanadium complex or $Mn_2(CO)_{10}$ as the homogeneous catalyst [10,11]. Also, several nickel complexes were found to be very efficient to the aliphatic cleavage of C—C bond at ambient conditions [12–16]. Furthermore, Schwarz et al. employed a simple $CeCl_3$ as catalyst to achieve the cleavage of vicinal diol under blue light in air [17]. Very recently, the green and aerobic oxidative cleavage process of vicinal diol was successfully performed using carbon nitrides as catalyst in micellar medium under the visible light [18].

Furthermore, to resolve the low stability and poor recyclability of homogeneous metallic catalyst, the heterogeneous solid catalysts were paid great attentions owing to their excellent stability and good recyclability. In particular, transition metal oxides represent the main heterogeneous catalysts used in industry [19]. In 2017, Anasta's group [20,21] discovered that a mixed oxide of Na and Mn (Na-Mn-LMO) or Mn oxide from biomass of Mn-hyperaccumulating plant could efficiently promote the oxidative carbon-carbon bond cleavage of 1, 2-diols in 1-butanol. As a result, different aldehydes or ketones can be prepared using K_2CO_3 as the additive and using molecular oxygen as an oxidant under the suitable conditions. Luo et al. [22] studied the oxidative cleavage of 1, 2-diols with an atomically dispersed Co-based heterogeneous catalyst supported on ***N***-doped carbon materials, in which several reactants could be efficiently transformed to corresponding aldehydes in

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https://doi.org/10.1016/j.catcom.2021.106305
Received 6 November 2020; Received in revised form 9 February 2021; Accepted 12 March 2021
Available online 16 March 2021
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the presence of K_2CO_3 as additive. Moreover, Solmi et al. [23] used the Au/MgO catalyst for the oxidative cleavage of trans-1, 2-cyclohexanediol with air, in which a close to 70% selectivity of adipic acid was attained under the optimal conditions.

The application of nickel element recieved substantial attentions because of its rich content and low toxicity [24]. Particularly, nickel oxide (NiO) is a solid material with ferromagnetic properties and its Neel temperature is about 523 K. Correspondingly, the NiO has some unique electrical, magnetic, and optical properties that make it greatly popular in traditional field [25]. Thus, the NiO is often employed as the sensor [26], the photoelectrochemical device [27–29], the battery [30] and electro chromic film [31,32]. Also, the supported NiO nanosheet is considered as the supercapacitor [33]. However, use of nickel oxide as the efficient heterogeneous catalyst is relatively inadequate, except that the K/Ca/Ni oxide system was investigated on the oxidative coupling process of methane [34].

In this communication, the simple and low-cost black nickel oxide (NiO_x) was employed as the catalysts to perform the oxidative cleavage of 1, 2-diols in aliphatic alcohols. Based on the experimental results, it was found that, for the oxidation of 1-phenyl-1, 2-ethanediol, a 97.5% conversion in 96.7% selectivity of benzaldehyde was obtained under 0.3 MPa of O_2 at 140 °C for 3 h. Nickel oxalate-derived catalyst is the most active for the oxidative C—C bond cleavage reaction. The relationship between the catalytic performance of NiO_x and material structure is discussed. The existence of Ni^{3+} should be crucial to the activity of catalyst. Furthermore, recycling experiments showed that the catalyst still retains a high activity even after being reused for five times.

2. Experimental section

2.1. Reagents and equipment

All reagents and chemicals used were of analytical grade unless otherwise specified. Nickel nitrate; oxalic acid, urea, 1-phenyl-1, 2-ethanediol, hydro benzoin, 2-methoxy-1, 2-diphenylethanone, 2-hydroxy-2-phenylacetic acid, Na_2CO_3, NH_3H_2O, NaOH, methanol, ethanol, n-propanol, i-propanol, n-butanol and n-pentanol are of analytical grade and used without further treatment. The standard samples of benzaldehyde and methyl benzoate are purchased from Alfa Aesar.

The measurement of X-ray diffraction (XRD) was performed by a diffractometer with Cu Ka radiation and was collected from 5 to 80° [2θ] with a resolution of 0.02 degree. The surface morphology and particle size of solid catalysts were detected by scanning electron microscope (SEM) (JSM-6301F, JEOL) and transmission electron microscope (TEM) (JEM-2100, JEOL). The pore structures of heterogeneous catalysts were detected by the Micromeritics ASAP2020M system. Fourier transform infrared (FT-IR) spectra were recorded on a Nicolet Nexus spectrometer in the 400–4000 cm^{-1} range. The oxidation states of nickel in the catalysts were tested by H_2 TPR technique. The analysis of the product was carried out on the Agilent 6890/5973 Gas Chromatograph-Mass Spectrometer (GC–MS) instrument.

2.2. The preparation of the NiO-H and NiO-C solid catalysts

Approximately 5.816 g of $Ni(NO_3)_2$ was dissolved in 20 mL of deionized water. Under stirring, the 0.1 M NaOH solution was slowly added until the pH value retains in the range of 8–9; then, the mixture was further stirred at 60 °C for 4 h. After being cooled to room temperature, the solid product was obtained by the centrifugation. Thereafter, the solid was washed by the distilled water and anhydrous ethanol several times, and then dried under vacuum. Finally, the NiO-H was prepared through being calcined at 300 °C for 2 h.

The preparation of NiO-C catalyst is similar to that of NiO-H catalyst but uses Na_2CO_3 solution instead of NaOH solution.

Table 1
The oxidative C—C bond cleavage of **1** with different catalysts [a].

Entry	Catalyst	Conversion (%) [b]	Product Distribution (%) [b]	
			2	**3 + 4**
1	NiO-H	14.0	98.7	1.3
2	NiO-C	34.7	99.6	0.4
3	NiO-U	49.4	99.8	0.2
4	NiO_x	61.1	98.5	1.5
5	NiO-250	11.7	100	–
6	NiO-400	27.7	99.8	0.2
7	NiO-500	4.6	98.1	1.9
8	NiO-300[c]	1.7	100	–
9	NiO-300-A[d]	29.3	97.5	2.5
10	NiC_2O_4	2.1	100	–
11	no	0.3	100	–
12[e]	NiO_x	97.5	96.7	3.3

[a] Reaction conditions: 0.2 g of **1**, 0.05 g of nickel oxide catalyst, in 20 mL methanol, under 0.3 MPa of oxygen, at 100 °C, for 4 h. [b] The data were attained by GC using the internal standard technique. [c] The NiO-300 catalyst is prepared *via* the calcination of NiC_2O_4 under N_2 300 °C. [d] NiO-300-A is obtained by continuous treatment of the catalyst in entry 8 under air atmosphere at 300 °C. [e] The reaction is performed at 140 °C for 3 h.

2.3. The preparation of the NiO-U catalyst

About 4.9772 g of nickel acetate was dissolved in a solution of 30 mL of ultrapure water and 30 mL of ethylene glycol. Under stirring, the urea (6.0 g) and polyvinylpyrrolidone (PVP, 0.13 g) were added to above solution. Then, the mixture was transferred to Teflon-lined autoclave for hydrothermal crystallization at 180 °C for 5 h. After being cooled to room temperature, the solid product is obtained through the centrifugation. Thereafter, the solid was washed by the distilled water and anhydrous ethanol several times, and then dried under vacuum. Finally, the NiO-U was prepared through calcination at 300 °C for 2 h.

2.4. The preparation of NiO_x catalyst

About 5.816 g of Ni $(NO_3)_2$ was dissolved in 20 mL of water; then, the 25% ammonia water was dropped until pH = 8.0 (Solution A); In addition, about 3.78 g of oxalic acid was dissolved in 20 mL of distilled water (Solution B). Next, the solution B was slowly added into solution An under stirring to pH 7.0. The obtained mixed solution was stirred at 60 °C for 4 h. After cooling to the room temperature, the solid products were attained through the centrifugation, then, the product was dried under a vacuum condition. Finally, the NiO_x material was prepared after being calcined at 300 °C for 2 h.

2.5. General procedure for selective oxidative cleavage of C—C bond

The oxidative carbon-carbon bond cleavage reaction of substrate was performed in a 120 mL stainless-steel autoclave with the magnetic stirring. The general procedure for oxidative transformation of 1-phenyl-1, 2-ethanediol is described as follows: 0.200 g of reactant, 0.050 g of NiO_x catalyst, and 20 mL of methanol were added to the autoclave, respectively. The atmosphere in the reactor was exchanged by pure oxygen for thrice. Then 0.3 MPa of dioxygen was charged into the reactor after being sealed. Next, the temperature was raised to 140 °C, and was kept for 3 h. After reaction, the mixture was slowly cooled and the excessive gas was discharged. Finally, the solid NiO_x catalyst was separated by the filtration, and the products were analyzed by the Gas Chromatography (GC) with a hydrogen ion flame detector using the internal standard technique.

3. Results and discussion

Initially, aerobic oxidative cleavage of 1-phenyl-1, 2-ethanediol (**1**) was used as the model reaction to investigate the activity of different

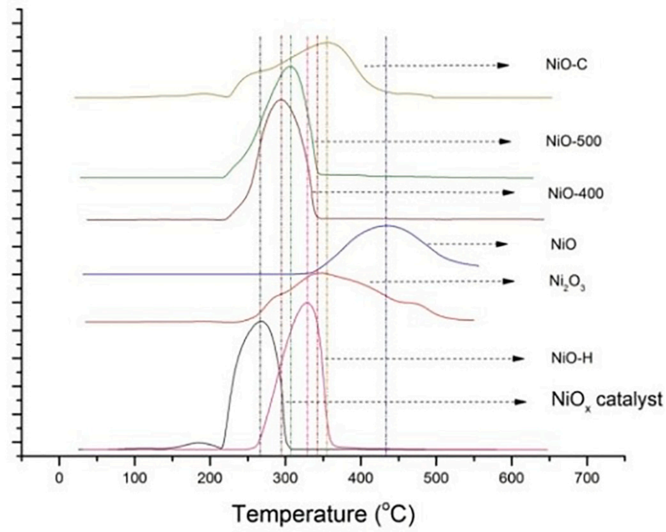
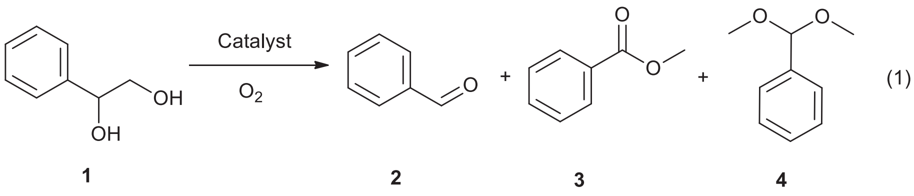


Fig. 1. The H_2-TPR detection of nickel oxides prepared using different methods.

catalyst (Eq. 1), in which the main product is benzaldehyde (**2**) and the obtained by-products include methyl benzoate (**3**) or (dimethoxymethyl)benzene (**4**) during reaction. The experimental results are summarized in Table 1.



(1)

In the presence of NiO-H catalyst (prepared using sodium hydroxide as precipitant), a 14.0% conversion of **1** in 98.7% selectivity of **2** was obtained in methanol solvent (entry 1). When NiO-C (prepared using sodium carbonate as precipitant) or NiO-U (prepared using urea as the precipitant) was used as catalyst, a 34.7% or 49.4% conversion of **1** is attained (entries 2 and 3). Furthermore, the NiO_x (originated from calcination of NiC_2O_4 at 300 °C) exhibits very high catalytic activity, in which the conversion of **1** can arrive at 61.1% under similar conditions (entry 4). These indicate that the synthesis processes of nickel oxide and the corresponding precursors are closely related to the catalytic performance and that nickel oxalate-derived catalyst is the most active for oxidative C—C bond cleavage reaction.

In addition, the effect of calcination temperature was further investigated for this process. As a result, it is found that 11.7%, 27.7 or 4.6% conversion of **1** was obtained when NiO-250 (prepared from calcination of NiC_2O_4 at 250 °C), NiO-400 or NiO-500 was employed as the catalyst (entries 5, 6 and 7), respectively. These results indicated that the NiC_2O_4 being calcined at 300 °C is beneficial to promote reaction. Conversely, when the NiO is prepared *via* the calcination of NiC_2O_4 under nitrogen atmosphere, the activity remains substantially low and the conversion of **1** is about 1.7% (entry 8). In the following, after the above NiO is further treated under air, the conversion of **1** can reach 29.3%, which demonstrates that the generation of Ni^{3+} is responsible for the oxidative cleavage process (entry 9). As comparison, only 2.1% conversion of **1** is attained with NiC_2O_4 as the catalyst (entry 10). The blank experiment shows that less than 1% conversion (about 0.3%) of **1** is obtained in the absence of any catalyst (entry 11). Notably, a 97.5% conversion of **1** with 96.7% selectivity of **2** is attained at 140 °C for 3 h (entry 12), which

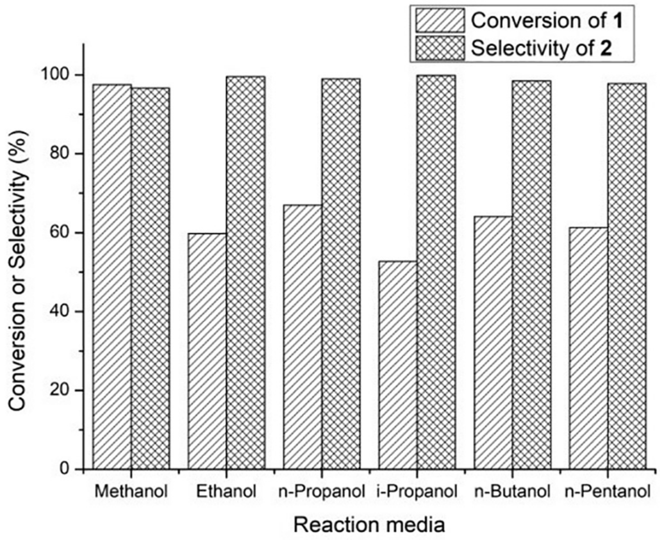


Fig. 2. Influence of solvent in oxidative C—C bond cleavage reaction (0.20 g of **1**, 0.05 g NiO_x, 20 mL solvent, under 0.3 Mpa of O_2, at 140 °C, for 3 h).

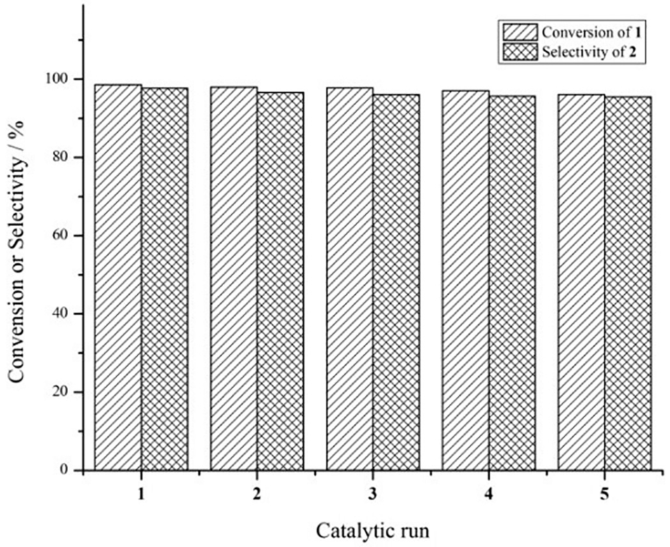


Fig. 3. Recycling of NiO_x on the oxidative C—C bond cleavage of **1** Reaction conditions: 0.20 g of **1**, 0.05 g catalyst, 0.005 g K_2CO_3, in 20 mL methanol, under 0.3 Mpa of O_2, at 140 °C, for 3 h).

Table 2
The oxidative C—C bond cleavage of different 1, 2-diols with the NiO_x catalyst [a].

Entry	Reactants	Conversion (%) [b]	Product distribution (%) [b]	
			Aldehyde	Others
1	(1*R*,2*S*)-1,2-diphenylethane-1,2-diol	99.0	98.8	1.2
2	(1*R*,2*R*)-1,2-diphenylethane-1,2-diol	96.7	>99.0	–
3	(1*S*,2*S*)-1,2-diphenylethane-1,2-diol	96.5	>99.0	–
4	1,2-bis(4-fluorophenyl)ethane-1,2-diol	92.1	>99.0	–
5	1,2-bis(4-bromophenyl)ethane-1,2-diol	94.3	96.5	3.5
6	1,2-bis(2-chlorophenyl)ethane-1,2-diol	98.9	>99.0	–
7 [c]	1, 2-di-p-tolylethane-1,2-diol	91.4	>99.0	–
8	1-(pyridin-2-yl)-2-(pyridin-3-yl) ethane-1,2-diol	96.9	98.3	1.7
9 [c]	2-methoxy-1, 2-diphenylethanone	>99.0	98.5 [d]	1.5
10	2-hydroxy-2-phenylacetic acid	>99.0	98.2 [e]	1.8

[a] Reaction conditions: 0.10 g of reactant, 0.05 g of the NiO_x catalyst, in 20 mL of methanol, under 0.3 MPa of oxygen, at 100 °C, for 3 h. [b] The data were attained by GC using the internal standard technique. [c] The reaction is performed at 140 °C. [d] The main product is methyl benzoate. [e] The obtained main product is (dimethylhydroxy)benzene in the methanol solvent.

indicates that the conversion of **1** is obviously improved and that the selectivity of **2** still maintains unchanged almost when the temperature is elevated.

The reduction behavior and oxidative states of nickel oxides were characterized by temperature-programmed reduction (H_2-TPR) measurement, and the results are given in Fig. 1. Generally, the reduction peaks should be related to the transformation of Ni_2O_3 to NiO and the following the reduction of NiO to Ni during reaction [35]. The corresponding temperature may respond the characters of nickel oxides. If the temperature of peaks is lower, the corresponding nickel oxide nanomaterial is more active on the catalytic reaction. On the basis of the obtained experimental results, it can be observed that the strongest peaks of commercial NiO and Ni_2O_3 are located at 430 °C and 340 °C, respectively. In addition, both of them belong to the wide peaks. Furthermore, the peaks of the as-prepared nickel oxides including NiO_x, NiO-H, NiO-C, NiO-400 and NiO-500 are located at between 250 and 400 °C, where most of them are the relatively sharp peaks except that of the NiO-C material. In particular, the reduction temperature of NiO_x catalyst is between 210 and 305 °C and the temperature of the corresponding peak is at 265 °C. These demonstrated that NiO_x still remains very active and easily activated under the suitable conditions.

Furthermore, the selective oxidative C—C bond cleavage reaction of **1** with NiO_x catalyst was further investigated in various aliphatic alcohols. As shown in Fig. 2, a 59.8% conversion of **1** and 99.6% selectivity of **2** was obtained in ethanol, in which the conversion of the reaction is a little lower than that in methanol. Otherwise, when the selective oxidative cleavage process was performed in *n*-propanol or *i*-propanol solvent, a 67.0% or 52.7% conversion of **1** was attained, where the selectivity of **2** still remained more than 99% during the reaction. In addition, a 64.1% or 61.3% conversion of **1** was achieved when the *n*-butanol or *n*-pentanol was employed as the solvent, which is similar to that using ethanol as solvent in this reaction. These results clearly showed that the methanol should be a preferable solvent for the selective oxidative C—C bond cleavage of 1, 2-diols.

To study the stability of NiO_x catalyst system, the recycling experiments were performed for the oxidative C—C bond cleavage reaction of **1** in the presence of molecular oxygen. After the first catalytic run, the black solid NiO_x catalyst is filtered and washed by anhydrous ethanol for more than thrice; next, the obtained sample is further dried at 80 °C for 12 h in a vacuum oven and then used as catalyst in the following reactions. As shown in Fig. 3, the solid NiO_x catalyst still exhibited a high activity and good selectivity even after being employed for four times. The conversion of **1** is still over 95% and the selectivity of **2** remains nearly unchanged. The experimental results indicate that the NiO_x catalyst can be recycled and reused during the oxidative C—C bond cleavage processes.

Finally, the selective oxidation processes of different substrates were investigated with NiO_x catalyst. The obtained experimental data from the oxidative cleavage processes are given in Table 2. As a result, it can be seen that more than 90.0% yields of corresponding aldehydes were obtained when different 1, 2-diols were employed, which further confirms that NiO_x catalyst is very high efficient for the oxidative C—C bond cleavage of vinical diols in the presence of oxygen. However, when the oxidation of 2-methoxy-1, 2-diphenylethanone was carried out with NiO_x catalyst in methanol, a 99.0% conversion with a 98.5% selectivity of methyl benzoate was attained. Meanwhile, 2-hydroxy-2-phenylacetic acid could be selectively converted to produce (dimethyoxymethyl) benzene in methanol. These results showed that NiO_x catalyst can promote oxidative cleavage of C—C bond of different reactants in the presence of molecular oxygen.

4. Conclusions

In summary, a novel and efficient oxidative cleavage process of 1, 2-diols and their derivatives with NiO_x catalyst was achieved. In the oxidation of compound **1** and hydrobenzoin, a 94.3% and 97.8% yield of benzaldehyde was respectively obtained. Thus, nickel oxalate derived catalyst is the most active for oxidative C—C bond cleavage reaction, compared to other catalyst precursors. Moreover, it was found that the calclination temperature plays a key role on the preparation of NiO_x with 300 °C being optimal. Furthermore, recycling experiments showed that the catalyst still maintains a high activity even after being reused five times. This provides a novel and efficient approach for the catalytic valorization of biomass and biomass-derived platform compounds.

Declaration of Competing Interest

For the paper titled as “A novel selective oxidative cleavage of C-C bond mediated by black nickel oxide in the presence of molecular oxygen” submitted to the Catalysis Communications journal, 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.

Acknowledgements

This work is financially supported by the National Natural Science Foundation of China (No. 21878235 and 21336008).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.catcom.2021.106305.

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