



Short communication

In situ grown palladium nanoparticles on polyester fabric as easy-separable and recyclable catalyst for Suzuki-Miyaura reaction

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ABSTRACT

Palladium nanoparticles supported on low-melting polyester (Pd/LMPET) fabric were prepared through a microwave irradiation assisted method. In this way, *in situ* growth of Pd nanoparticles onto an easy to handle material was initiated and proceeded. The results of the characterization revealed that the palladium nanoparticles were well-dispersed on the surfaces of the polyester fibers. The Pd/LMPET fabrics were then employed in the Suzuki-Miyaura coupling. They exhibited excellent catalytic activity in ethanol/water under air atmosphere at 50 °C. Importantly, the Pd/LMPET fabrics could be separated from reaction mixture conveniently and they can still maintain good activity after 8 cycles without Pd leaching.

1. Introduction

Metal catalysis is often a pivotal part in fine chemical and pharmaceutical synthesis [1,2]. In this regard, palladium-catalyzed cross-coupling reactions have attracted widespread attention in the construction of carbon-carbon bonds [3,4], and the Suzuki-Miyaura cross-coupling reaction is one of the most effective way to form various bi-phenyls and its derivatives in organic synthesis [5,6]. As originally developed, homogeneous Pd complexes were employed as efficient catalysts. However, homogeneous palladium catalysts also face many challenges, including large-scale use, product purification (Pd removal), and tedious separation and recycling. Moreover, in most Pd-catalyzed homogeneous catalyst systems, the reactions require organic solvents, inert gas protection and elevated temperatures. Such disadvantages make homogeneous catalytic synthesis uneconomical and environmentally unfriendly. It is an inevitable trend to develop green and sustainable catalytic systems. Therefore, heterogenization of homogeneous catalysts on solid supports or the development of novel heterogeneous catalysts are considered to overcome the inherent disadvantages of homogeneous catalysts.

Presently, the immobilization of palladium catalysts on various organic and inorganic supports has been extensively studied, including

metal oxides [7], silica [8,9], zeolite [10], Fe₃O₄ [11], microbeads [12], polymers [13,14], cellulose [15,16], carbon materials [17], carbon nitride [18], metal organic frameworks [19,20], and covalent organic frameworks [21,22], displaying improved environmental friendliness, stability and reusability. However, some of these heterogeneous catalytic systems still have disadvantages such as complex preparation processes, catalyst leaching, relatively long reaction times and higher reaction temperatures. The best solution that combines simple, inexpensive manufacturing of efficient catalysts with easy-separable and effective recycling is still difficult to achieve. Recently, a low-melting polyester fiber (LMPET) with a sheath-core structure has been developed [23]. This kind of fiber was already proven useful as catalyst support, in which the sheath polyester with the melting point of about 120 °C can be used for binding with the target catalyst, and the core polyester with the melting point around 250 °C provides strong mechanical support. In detail, Lu et al. reported that g-C₃N₄/LMPET can be used to degrade organic pollutants and transform phenylboronic acid to phenol [23,24], where g-C₃N₄ powders were distributed over the surface of LMPET via dip-nip adsorption and thermal bonding. This heterogeneous catalyst is easy-separable and can be recycled for many times. Consequently, this material should be extended for the deposition of catalytic active particles for application in organic synthesis.

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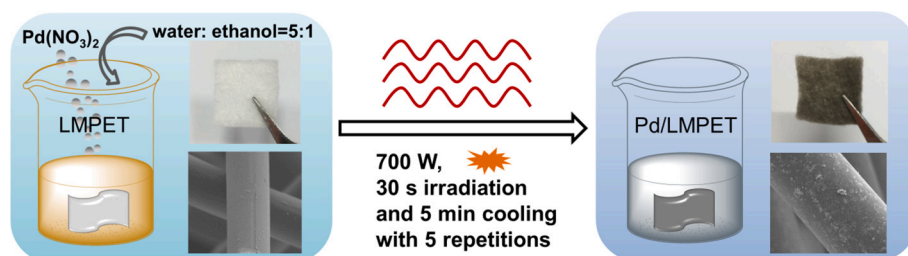


Fig. 1. Schematic illustration of synthesis of Pd/LMPET.

The microwave heating process is to increase the temperature of the heated material through the high-frequency reciprocating movement of the dipole molecules inside the heated body, which is completely different from other conventional heating methods. The application of the microwave irradiation assisted method has been reported for the preparation of different heterogeneous single-atom metal catalysts [18,25–27] and metal nanoparticles catalysts [28,29]. There is no reducing agent in the preparation process, and the preparation method is simple and efficient. Inspired by the above, we intended to use a microwave irradiation assisted method to load Pd onto LMPET fibers. The microwave method was used to heat the low-melting polyester fiber, where the sheath polyester can produce effective physical adhesion. In the following we report on an efficient, economical, recyclable, and easy-separable composite consisting of palladium nanoparticles supported on polyester fabric (Pd/LMPET) prepared *via* such a microwave method. The material was characterized by various analytical techniques, such as ICP-OES, FESEM, EDX, XPS ATR-IR, and XRD. It was exploited in an organic model reaction, namely in the Suzuki-Miyaura

cross-coupling.

2. Experimental

2.1. Preparation of Pd/LMPET

Polyester fabric (LMPET, 481.2 mg) was first cut into $2\text{ cm} \times 2\text{ cm}$ pieces and then immersed in H_2O (50 mL) and ethanol (10 mL). Pd (NO_3)₂ solution containing 4.812 mg in 2 mL of deionized water was added to it. Afterwards, the resulting mixture was placed in a microwave reactor (Sharp R204S Solo microwave oven), with a cyclic program of 30 s irradiation and 5 min cooling with 5 repetitions using a power of 700 W. Afterwards, the LMPET fabric was collected, washed with ethanol and distilled water, and dried at $60\text{ }^\circ\text{C}$. This sample was named as 1Pd/LMPET (where Pd(NO_3)₂ content in the reaction mixture was 1% related to LMPET; the theoretical Pd content in the composite is about 0.424% related to LMPET; ICP-OES analysis reveals the Pd amount of 0.187 wt%). For comparison, 0.1Pd/LMPET (0.1% Pd(NO_3)₂ related to

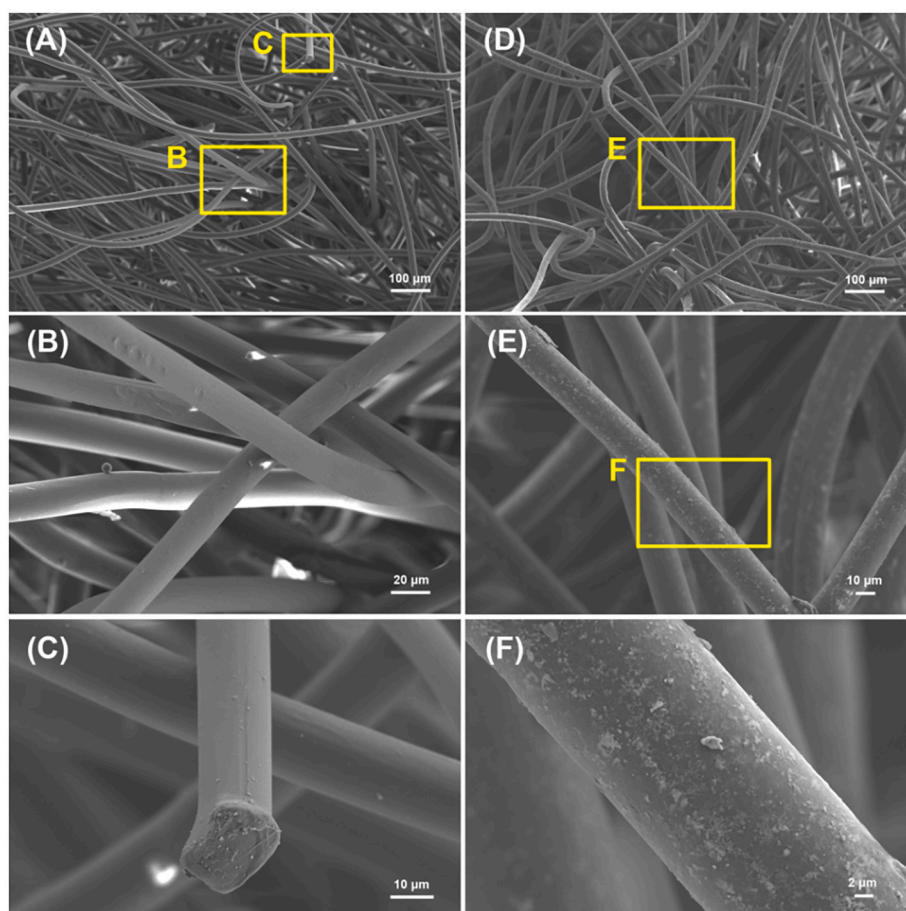
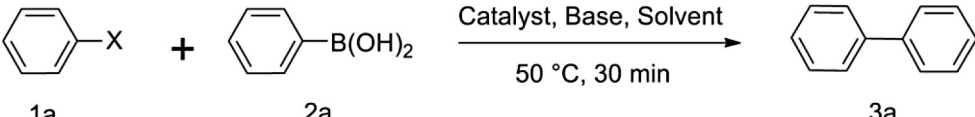


Fig. 2. (A), (B) and (C) SEM images of the surface of untreated LMPET; (D), (F) and (E) SEM images of the surface of 1Pd/LMPET.

Table 1
Optimization of reaction conditions.^a



Entry	X	Catalyst	Solvent	Base	Yield (%) ^b
1	Br	1Pd/LMPET	EtOH	K ₂ CO ₃	49
2	Br	1Pd/LMPET	MeOH	K ₂ CO ₃	27
3	Br	1Pd/LMPET	Toluene	K ₂ CO ₃	NR
4	Br	1Pd/LMPET	DMF	K ₂ CO ₃	NR
5	Br	1Pd/LMPET	H ₂ O	K ₂ CO ₃	6
6	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	100 (98 ^c)
7	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	Na ₂ CO ₃	88
8	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	NaHCO ₃	66
9	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	KHCO ₃	60
10	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	Et ₃ N	65
11	Br	1Pd/LMPET	TBA: H ₂ O = 1:1	K ₂ CO ₃	53
12	Br	LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	NR
13 ^d	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	89
14 ^e	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	94
15 ^f	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	42
16 ^g	Br	1Pd/LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	92
17 ^h	Cl	1Pd/LMPET	EtOH: H ₂ O = 1:1	K ₂ CO ₃	7

^a Reaction conditions: 1a (0.5 mmol), 2a (0.75 mmol, 1.5 equiv), catalyst (100 mg, 0.36 mol% palladium with respect to aryl halide), base (3.0 equiv), solvent (4 mL), 50 °C, 30 min.

^b GC yields were determined by using hexadecane as the internal standard.

^c Isolated yield is in the parenthesis.

^d 2a (0.6 mmol, 1.2 equiv).

^e 10 min.

^f Room temperature.

^g Room temperature, 2 h.

^h Reaction time 3 h.

LMPET), 0.5Pd/LMPET (0.5% Pd(NO₃)₂ related to LMPET) and 2Pd/LMPET (2.0% Pd(NO₃)₂ related to LMPET) were also prepared under the same experimental conditions, and ICP-OES analysis of 0.5Pd/LMPET and 2Pd/LMPET was shown in Table S1, which reveals the Pd amount of 0.136 wt% and 0.309 wt%, respectively.

2.2. General procedure for Suzuki-Miyaura cross-coupling reaction

In a typical Suzuki-Miyaura cross-coupling experiment, aryl halide (0.5 mmol), phenylboronic acid (0.75 mmol), base (1.5 mmol) and 100 mg Pd/LMPET were mixed in the solvent (4 mL) and stirred at 50 °C for a desired time. All reactions were carried out under air atmosphere. The final products of the reaction were extracted with ethyl acetate and the yield was analyzed by GC.

3. Results and discussion

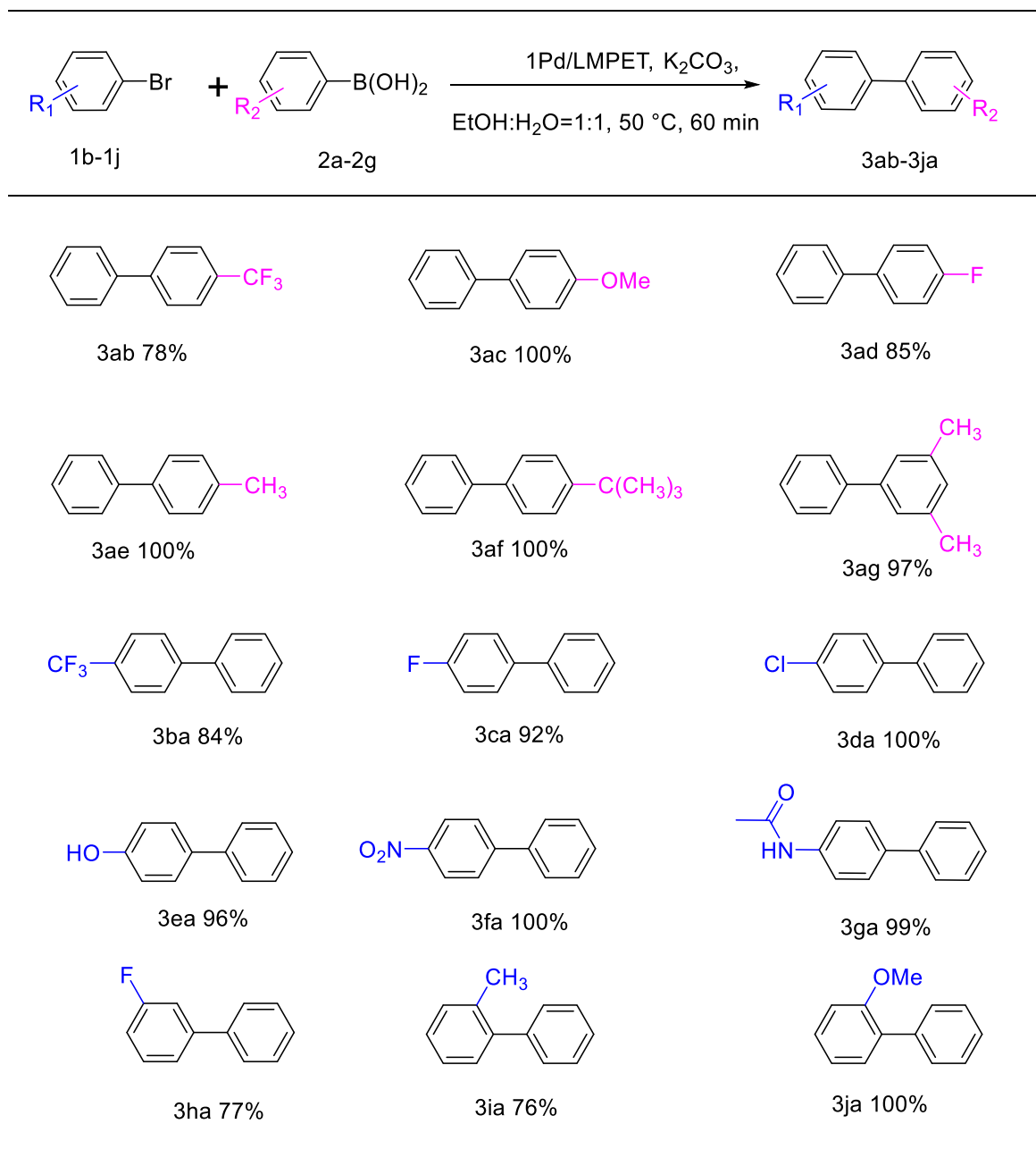
3.1. Description of catalysts

The preparation process of the Pd/LMPET fabric is depicted in Fig. 1. By simple microwave heating of a Pd(NO₃)₂ solution, Pd particles were deposited on the PET fibers resulting in an easy to handle fabric with catalytic functionality. The morphologies of as-prepared catalysts were characterized by SEM and the results are shown in Fig. 2(A-F). Fig. 2(A-C) indicate that the surface of pure LMPET is smooth. After the microwave synthesis, SEM images show deposited Pd nanoparticles

distributed over the surface of the morphologically unchanged LMPET (Fig. 2(E and F)). As can be seen from Fig. 2(C and F), the fiber diameters of LMPET and Pd/LMPET are both around 18 μm, which additionally demonstrates that the support material remains unchanged during its surface decoration with Pd. An overview of the Pd/LMPET fabric before and after usage is shown in Fig. S1 and Fig. S2, where the overall morphology of the fabric has not changed. The distribution of elements C, O and Pd in the 1Pd/LMPET and 1Pd/LMPET after 8 cycles were further characterized by EDX elemental mapping (Fig. S3(A-H)). LMPET is mainly composed of carbon and oxygen. As shown in Fig. S3D, Pd is evenly distributed on the fiber surface before reaction, which again confirms the excellent dispersity of Pd on the LMPET via microwave irradiation method. After 8 cycles, Pd is still present on the fiber surface, indicating that Pd is stably attached on the LMPET surface. However, there are some changes of the Pd found on the fiber surface. The Pd is less evenly distributed or agglomerated indicated by surface imaging of the spent catalyst fiber (Fig.S2). We also found that most fibers keep their morphology after 8 cycles but show crack formation to a certain extent. The appearance of cracks may be ascribed to the multiple cleaning and drying during the cycles. Fig.S4 is a typical TEM image of Pd/LMPET, it shows Pd is present in the form of nanoparticles.

Surface elemental composition and the coordination states of Pd in the Pd/LMPET were investigated by X-ray photoelectron spectroscopy (XPS). As shown in Fig. S5A, the XPS spectrum of 1Pd/LMPET present four peaks (at 335.4, 337.1, 340.7 and 342.5 eV) which were attributed to Pd 3d_{5/2} and 3d_{3/2} [30,31]. The peaks appearing at 335.4 and 340.7

Table 2
Effect of substituent groups on 1Pd/LMPET catalyzed Suzuki-Miyaura reaction.



eV were assigned to metallic Pd (Pd^0). Two peaks observed at 337.1 and 342.5 eV are the features of divalent Pd (Pd^{2+}). The results indicate that the palladium particles are partially reduced on the LMPET surface. Additionally, the presence of Pd^{2+} may be attributed to the incomplete reduction of Pd^{2+} during the preparation process, or the oxidizability of the obtained metal Pd^0 in the air atmosphere. After 8 cycles, the metallic Pd^0 and Pd^{2+} still exist on the catalyst, which indicates that Pd active species on Pd/LMPET are stable. Additionally, the Pd content in the Pd/LMPET fabric after the 8th cycle was determined by ICP-OES analysis to be 0.182 wt%, which is basically consistent with the Pd content of the catalyst before use, indicating that the coupling reaction catalyzed by Pd/LMPET fabric was not affected by leaching of the Pd species.

ATR-FTIR spectra as well as X-ray diffraction (XRD) patterns of the support as well as of fresh and spent catalysts were recorded. In ATR-

FTIR only characteristic peaks of the polyester were detected, which proves that the structure of polyester fiber is stable (Fig. S6). In XRD, the diffraction pattern of Pd could not be found, which may be caused by the low content of Pd, but a broad signal from amorphous PET was detected (Fig. S7(A-D)).

3.2. Catalytic testings

Bromobenzene (1a) and phenylboronic acid (2a) were selected as model substrates for the optimization of reaction conditions with 1Pd/LMPET catalyst in the Suzuki-Miyaura coupling reaction. In view of recent progress in Suzuki-Miyaura reaction, the reaction was firstly performed by using K_2CO_3 as the base and ethanol (EtOH) as solvent under air atmosphere at 50 °C. In this initial experiment we could detect

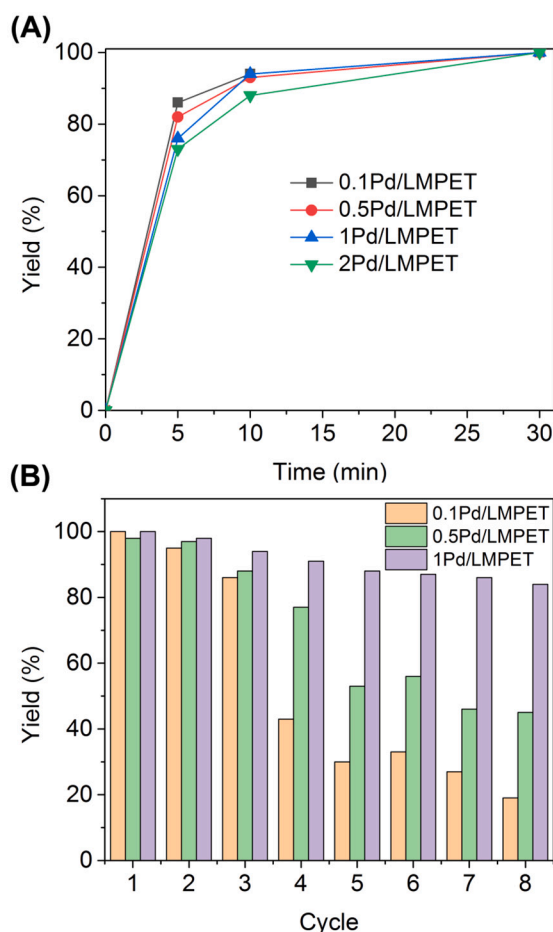


Fig. 3. (A) Yield-time curve obtained over Pd/LMPPET with different amounts of Pd; (B) Recycling performance of Pd/LMPPET with different amounts of Pd for Suzuki-Miyaura reaction. Reaction conditions: 1a (0.5 mmol), 2a (0.75 mmol), 1.5 equiv), catalyst (about 100 mg), base (3.0 equiv), solvent (4 mL), 50 °C.

our target coupling product 3a with 49% GC yield (Table 1, entry 1). Next we evaluated the influence of various solvents. Other organic solvents such as methanol (MeOH), toluene and dimethylformamide (DMF) resulted in lower reaction performance or did even not react after 30 min (Table 1, entry 2–4). A lower yield of 6% for the target product 3a was also detected when water was used as the solvent (Table 1, entry 5). Considering the solubility of substrate and base, next a mixed solvent was chosen. The addition of water to ethanol tremendously increased the yield, giving 2a in a high yield of 100% (Table 1, entry 6). This significant improvement might be due to the good solubility of K_2CO_3 in water and the poor solubility of the product in ethanol/water mixture which shifts equilibrium and facilitates product formation [30]. We continued to explore the reaction with different bases. The bases such as Na_2CO_3 , $NaHCO_3$, $KHCO_3$, and triethylamine (Et_3N) gave the desired product in lower yields (Table 1, entry 7–10). In addition, the solvent mixture of tert-butanol/water ($v/v = 1/1$) reduced the catalytic performance (Table 1, entries 11). There was no product when the pure LMPET was used as the catalyst (Table 1, entry 12), which indicates the inertness of the support in the Suzuki-Miyaura reaction. Product 3a in 89% yield was obtained in the presence of 1.2 equivalents of phenylboronic acid (Table 1, entry 13). Reducing the reaction time from 30 to 10 min under remaining conditions of entry 6, the substrate could be transformed into the desired product in 94% yield (Table 1, entry 14). A satisfactory yield of 92% was obtained even at room temperature for 2 h reaction time applying the reactants from entry 6 (Table 1, entry 16), which indicates that this reaction can proceed under mild conditions.

However, in order to get a higher yield, 1.5 equivalents of phenylboronic acid were used. Thus, the optimized reaction conditions were found to be in the presence of 0.5 mmol bromobenzene, 1.5 equivalents of phenylboronic acid and 3.0 equivalents of K_2CO_3 in ethanol/water ($v/v = 1/1$) under air atmosphere at 50 °C, which gave 3a in 98% isolated yield. Additionally, the catalyst system was evaluated with chlorobenzene under the optimized reaction conditions where only 7% yield could be obtained after 3 h.

Next we evaluated the substrate scope with a variety of arylhalides and phenylboronic acids at 50 °C for 60 min, and the results were summarized in Table 2. With 1Pd/LMPET fabric as catalyst, a wide range of arylboronic acids, bearing either electron-donating groups such as methyl, methoxy and tert-butyl or electron withdrawing groups such as fluoride, and trifluoromethyl can proceed smoothly to the corresponding biphenyls and delivers the desired products in good to excellent yields (3ab – 3ag). Gratifyingly, the 1Pd/LMPET fabric was also effective in most cases for bromobenzene and its derivatives, but relatively less effective using *o*-methyl bromobenzene as well as 1-bromo-3-fluorobenzene. The yield of the desired product was affected by the electronic effects and the steric hindrance of the substrate.

Besides, Fig. 3A shows the yield of the Suzuki-Miyaura reaction catalyzed by LMPET fabric with different Pd contents, which proves that the reaction requires only a low Pd content to obtain a high yield of the target product. In contrast, excessive palladium loading may increase the particle size and partial agglomeration of palladium as well as higher costs. Moreover, the catalytic reaction only requires short time, indicating that the catalyst has high catalytic efficiency. As illustrated in Table S2, the TON and TOF values of the Pd/LMPET are calculated for the Suzuki coupling reaction. 1Pd/LMPET can reach a conversion above 82% after only 5 min, with a turnover frequency (TOF) of $2736 h^{-1}$.

In order to examine the performance of Pd/LMPET catalysts, the results of the coupling of bromobenzene with phenylboronic acid of this work were compared with the previously reported Pd-based heterogeneous catalysts in the literature. As shown in Table S3, the catalyst works under milder reaction conditions and shorter reaction time. The yield and TOF are higher than those in most references, or maintains similar activity to the best recently published results. Compared with powder supports, such as $g-C_3N_4$ and Ce_xO_y , LMPET fabric is a simple, easy to scale up material which can be easily removed. Meanwhile, the Pd/LMPET fabric has the advantages of simple preparation process, low cost, and, most importantly, easy separation, which is superior to the previously reported methods.

In the light of green chemistry, the separation and recyclability of precious metal catalysts is a critical factor in industrial applications. As shown in Fig. 3, the Pd/LMPET fabrics with different amount of Pd were reused eight times. 1Pd/LMPET fabric show the best recycling performance and the yield can still maintain over 80% after eight cycles, indicating its better recycling performance in the Suzuki-Miyaura reaction than that of 0.1Pd/LMPET and 0.5 Pd/LMPET. There is usually a slight decrease in yield, which is due to the leaching of Pd [32] and the agglomeration of Pd nanoparticles [33]. Here, it should be ascribed to the loss of fibers of the Pd/LMPET fabric caused by magnetic stirring ($\sim 22\%$ after 8 cycles, Fig. 3B) and the agglomeration of partial palladium nanoparticles in the recycle processes (Fig.S2). Importantly, compared with other heterogeneous powder catalysts, the Pd/LMPET fabric can be separated from the reaction solvent without centrifugation or other external force, which is very beneficial to industrialization but also for screening processes. Additionally, hot filtration was tested to determine catalyst nature, which was carried out on the coupling reaction of bromobenzene with phenylboronic acid under the optimal reaction conditions [34,35]. After 3 min reaction time, when 42% yield was obtained, the Pd/LMPET was removed from the reaction medium and the process was monitored for another 20 min. No improvement was detected in the product yield. This observation confirms the heterogeneity of Pd/LMPET.

4. Conclusion

We have used a microwave irradiation assisted method for the generation of Pd/LMPET fabric using Pd(NO₃)₂ as precursor and aqueous/ethanol as medium for *in situ* growth of Pd nanoparticles on the LMPET fibers. The material was successfully applied as catalyst in the Suzuki-Miyaura reaction of bromobenzene with phenylboronic acid, exhibiting high efficiency with low Pd loading, mild reaction conditions, and high yields. More importantly, it demonstrated good recycling ability and the unique advantage of convenient catalyst separating after several cycles. The morphology and structure of the used Pd/LMPET fabric was characterized indicating a further demand on optimization strategies. Predictably, this work can provide a general way on the design and preparation of different metal catalysts onto easy-separable polymeric materials.

Declaration of Competing Interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.catcom.2021.106328>.

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