



WATER EXTRACT OF ONION PEEL ASH: AN EFFICIENT GREEN CATALYTIC SYSTEM FOR THE SYNTHESIS OF ISOINDOLINE-1,3-DIONE DERIVATIVES

(Air Abu Kulit Bawang: Sistem Pemangkin Katalitik Hijau yang Effisien dalam Sintesis Terbitan Isoindolina-1,3-dion)

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Abstract

Isoindoline-1,3-dione derivatives are important organic compounds associated with various interesting applications such as in the fields of material science, medicine, important building blocks and so on. Traditionally, the synthesis of isoindoline-1,3-dione derivatives are achieved using harmful and toxic catalysts, such as concentrated sulphuric acid, triphenylphosphine, ionic liquid [bmim][BF₄] or phthalimide-N-sulfonic acid. Thus, the development of a greener catalytic system for the synthesis of isoindoline-1,3-dione derivatives is highly sought after. In this work, an efficient synthesis of isoindoline-1,3-dione derivatives using the Water Extract of Onion Peel Ash (WEOPA) method is described. The new approach provides several advantages including the non-usage of external acids, recyclable catalytic system, cheap and the yield of the synthesized products were obtained in moderate to good yields by reacting various anilines with phthalic anhydride. The current improved method is scientifically important, due to it avoid the use of environmental harmful reagents and at the same time provides an alternative method for bio-waste management.

Keywords: water extract of onion peel ash, isoindoline-1,3-dione, recyclable catalytic system, anilines, phthalic anhydride

Abstrak

Terbitan isoindolina-1,3-dion adalah sebatian organik yang penting dan dikaitkan dengan pelbagai aplikasi yang menarik, seperti dalam bidang sains bahan, perubatan, blok bangunan penting dan sebagainya. Secara tradisinya, sintesis derivatif isoindolina-1,3-dion dicapai dengan menggunakan pemangkin berbahaya dan beracun, seperti asid sulfurik pekat, trifenilfosfin, cecair ionik [bmim][BF₄] atau asid fitalimid-N-sulfonik. Oleh itu, pembangunan sistem pemangkin yang lebih hijau untuk sintesis derivatif isoindolina-1,3-dion sangat diperlukan. Dalam kerja ini, satu kaedah efisien dalam sintesis terbitan isoindolina-1,3-dion dengan menggunakan ekstrak air abu bawang dibincangkan disini. Kaedah baru ini memberikan pelbagai manfaat termasuk bebas daripada penggunaan asid luaran, sistem pemangkin kitar semula, murah dan hasil bagi produk sintesis yang didapati adalah sederhana hingga baik melalui tindak balas antara anilin-anilin dengan fitalik anhidrit. Kaedah yang ditambahbaik ini adalah penting secara saintifik, kerana ia mengelakkan penggunaan reagen berbahaya kepada alam sekitar dan pada masa yang sama menyediakan kaedah alternatif untuk pengurusan bio-sisa.

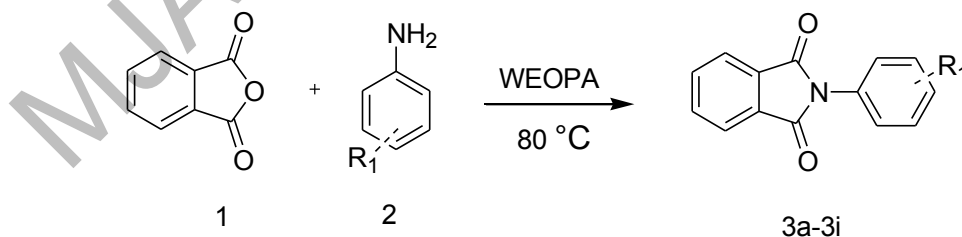
Kata kunci: ekstrak air abu bawang merah, isoindolina-1,3-dion, system pemangkin kitar semula, anilin, fitalik anhidrit

Introduction

Lately, there has been growing interest among chemists to develop simple and efficient green protocols for performing organic reactions due to the increase in environmental awareness worldwide [1-4]. The use of environmentally acceptable reagents, green solvents and biocatalysts are favorable, as it promote sustainable chemistry for synthesis processes conducted in both academic institutions and industries [5]. In particular, the use of aqueous media as green solvent has attracted the attention of the scientific community due to its environmental-friendly, cheap, benign solvent, non-flammability and readily available features, thus rendering its usefulness in the field of organic synthesis [6-8]. Furthermore, the use of water as a solvent in organic processes is promising and it is known to provide acceleration in the rate of chemical reactions as evidenced in a few recent literature [9-11].

At present, there has been growing awareness among synthetic chemists to utilize waste water, water extract of bio-wastes and vegetables juices to address and apply the concept of eco-friendly organic transformations. Among the main benefits identified from employing these green protocols include the biodegradable/recyclable nature and high catalytic activity of these green catalytic systems in organic transformations [12]. To date, numerous organic transformations have been carried out efficiently using these green and recyclable catalytic systems. Examples include the Suzuki-Miyaura reaction [13], synthesis of biaryls [14], Sonogashira reaction [15], Henry reaction [16], Dakin reaction [17] and so on. Based on a survey, the world generates approximately a million tons of bio-wastes monthly [18]. The rate of generation of bio-wastes is expected to escalate over time with the boom in world population [19]. From the perspective of green chemistry, unwanted wastes such as agricultural and food wastes are attractive resources as they are abundantly available and have the potential to become green catalytic systems in chemical processes.

Onions (*Allium cepa* L.) is one of the most important vegetable crops cultivated worldwide for culinary and other functional purposes [20]. In some developed countries, it is reported that more than 100,000 tons of onion waste are generated annually [21]. This bio-waste could adversely affect our environment and living organisms if it is sent directly to landfill without proper treatment [22]. As part of our continuous interest to develop green synthetic methodologies, we herein report on a simple, recyclable catalytic system and efficient protocol for the synthesis of isoindoline-1,3-dione derivatives using the Water Extract of Onion Peel Ash (WEOPA) (Scheme 1). Isoindoline-1,3-dione derivatives have found many attractive applications in material science [23], medicine [24], important building block [25] and so on. Traditionally, the synthesis of isoindoline-1,3-dione derivatives are achieved using harmful and toxic catalysts, such as the concentrated sulfuric acid [26] and triphenylphosphine [27]. Other methods are by using ionic liquid [bmim][BF₄] [28] and phthalimide-*N*-sulfonic acid [29], which entail the use of expensive reagents. The use of the WEOPA as a catalytic system to catalyze the synthesis of isoindoline-1,3-dione derivatives can reduce the environmental pressure exerted by bio-wastes. In addition, this work enables the development of a more sustainable synthesis that is of scientific significance and at the same time, overcome the aforementioned drawbacks.



Scheme 1. Synthetic route towards the preparation of isoindoline-1,3-dione derivatives in the presence of WEOPA

Materials and Methods

All chemicals and solvent were purchased from Sigma-Aldrich and Merck, were used without further purification. The ¹H and ¹³C-NMR experiments were recorded using CDCl₃ as solvent on a Bruker Avance III 400 spectrometer and the chemical shifts were given in parts per million (ppm). The GC-MS analyses were recorded using a

Shimadzu QP2010SE, equipped with a Supelco fused silica capillary column (30 m × 0.25 mm i.d., 0.25 mm film thickness). The metal content was characterized using Varian Vista Pro Inductive Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

Preparation of the water extract of onion peel ash

Onion waste was obtained from a nearby local waste disposal site at Kuala Nerus, Malaysia in March 2018. The outer layer and bulb of onion peel were separated, and the onion peel was washed with distilled water. The onion peels were then sliced into small pieces and air-dried for 2-3 days. The WEOPA catalytic system was prepared according to a previous method [12]. Initially, the dried peels (2.5 g) were burnt in the furnace at 500 °C for about 1 hour. The obtained ash was transferred into a 100 mL shake flask containing 25 mL of distilled water and the mixture was stirred for 1 hour. Subsequently, the mixture was filtered using Whatman filter paper No.1 and the resulting filtrate was termed as WEOPA.

General procedure for the synthesis of isoindoline-1,3-dione derivatives.

A 25 mL round bottom flask containing a mixture of phthalic anhydride (1.00 mmol) and aniline (1.00 mmol) was added with WEOPA (2 mL). The reaction mixture was stirred magnetically at 110 rpm and heated up to 80 °C. The progress of the reaction was monitored using Thin-Layer Chromatography (TLC). Upon the completion of the reaction, ethyl acetate (3 x 30 mL) was added into the mixture to extract the crude products. The organic layer was combined and dried with MgSO₄, before concentrated under reduced pressure. The crude product was purified over silica gel column chromatography with ethyl acetate/hexane (1:9) as its eluent. The aqueous recovered WEOPA was reused for the synthesis of other isoindoline-1,3-dione derivatives under identical condition.

Results and Discussion

Spectroscopic data of isoindoline-1,3-dione derivatives

2-phenyl isoindoline-1,3-dione **3a** ¹H-NMR (400 MHz, CDCl₃): δ 7.47-7.37 (m, 3H); 7.56-7.47 (m, 2H); 7.85-7.74 (m, 2H); 8.02-7.90 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 123.76, 126.61, 128.07, 129.19, 131.67, 131.82, 134.38, 167.34 ppm; GC-MS: C₁₄H₉NO₂, m/z 223.06 (M)⁺.

2-(*o*-tolyl) isoindoline-1,3-dione **3b** ¹H-NMR (400 MHz, CDCl₃): δ 2.21 (s, 3H); 7.20 (d, *J* = 7.5 Hz, 1H); 7.29-7.40 (m, 3H); 7.83-7.76 (m, 2H); 7.72-8.00 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 18.0, 123.82, 126.86, 128.83, 129.47, 130.60, 131.18, 132.11, 134.46, 136.59, 167.41 ppm; GC-MS: C₁₅H₁₁NO₂, m/z 237.08 (M)⁺.

2-(*m*-tolyl) isoindoline-1,3-dione **3c** ¹H-NMR (400 MHz, CDCl₃): δ 2.42 (s, 3H); 7.24-7.18 (m, 3H); 7.42-7.36 (m, 1H); 7.83-7.74 (m, 2H); 7.79-7.91 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.41, 123.66, 123.82, 127.31, 129.01, 129.13, 131.48, 131.82, 134.40, 139.21, 167.44 ppm; GC-MS: C₁₅H₁₁NO₂, m/z 237.08 (M)⁺.

2-(*m*-tolyl) isoindoline-1,3-dione **3d** ¹H-NMR (400 MHz, CDCl₃): δ 2.40 (s, 3H); 7.30 (s, 4H); 7.82-7.76 (m, 2H); 7.98-7.92 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.33, 123.65, 126.51, 129.02, 129.83, 131.88, 134.42, 138.20, 167.45 ppm; GC-MS: C₁₅H₁₁NO₂, m/z 237.08 (M)⁺.

2-(4-methoxyphenyl) isoindoline-1,3-dione **3e** ¹H-NMR (400 MHz, CDCl₃): δ 3.85 (s, 3H); 7.02 (d, *J* = 8.7 Hz, 2H); 7.33 (d, *J* = 9.1 Hz, 2H); 7.83-7.73 (m, 2H); 8.00-7.90 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 55.46, 114.46, 123.68, 124.31, 128.02, 131.83, 134.30, 159.29, 167.61 ppm; GC-MS: C₁₅H₁₁NO₃, m/z 253.07 (M)⁺.

2-(4-isopropylphenyl) isoindoline-1,3-dione **3f** ¹H-NMR (400 MHz, CDCl₃): δ 1.31 (d, *J* = 7.1 Hz, 6H); 3.06-2.90 (m, 1H); 7.37 (d, *J* = 1.2 Hz, 4H); 7.85-7.74 (m, 2H); 8.03-7.70 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 24.03, 34.01, 123.68, 126.67, 126.51, 127.27, 129.21, 131.91, 134.42, 148.94, 167.48 ppm; GC-MS: C₁₇H₁₅NO₂, m/z 265.11 (M)⁺.

2-(4-fluorophenyl) isoindoline-1,3-dione **3g** ¹H-NMR (400 MHz, CDCl₃): δ 7.20 (t, 2H); 7.83-7.36 (m, 2H); 7.80 (dd, *J* = 5.3 Hz, *J* = 3.0 Hz, 2H); 7.95 (dd, *J* = 5.3 Hz, *J* = 3.0 Hz, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 115.96,

116.31, 123.82, 127.63, 128.39, 128.43, 131.70, 134.54, 160.71, 163.23, 167.21 ppm; GC-MS: $C_{14}H_8FNO_2$, m/z 241.05 (M)⁺.

2-(4-chlorophenyl) isoindoline-1,3-dione **3h** 1H -NMR (400 MHz, $CDCl_3$): δ 7.40 (d, J = 8.7 Hz, 2H); 7.47 (d, J = 8.7 Hz, 2H); 7.85-7.74 (m, 2H); 8.01=7.88 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 123.90, 127.74, 129.31, 130.23, 131.57, 133.79, 134.62, 167.02 ppm; GC-MS: $C_{14}H_8ClNO_2$, m/z 257.02 (M)⁺.

2-(4-bromophenyl) isoindoline-1,3-dione **3i** 1H -NMR (400 MHz, $CDCl_3$): δ 7.36 (d, J = 8.7 Hz, 2H); 7.63 (d, J = 8.7 Hz, 2H); 7.80 (dd, J = 5.5 Hz, J = 3.1 Hz, 2H); 7.94 (dd, J = 5.5 Hz, J = 3.1 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.78, 123.86, 128.04, 130.76, 131.64, 132.28, 134.67, 166.94 ppm; GC-MS: $C_{14}H_8BrNO_2$, m/z 300.97 (M)⁺.

In the FT-IR analysis, the onion peels obtained from a local supermarket (Figure 1a) was compared to onion peels sampled from a local waste disposal site (Figure 1b). As shown in the FT-IR spectrum, the signals at 3393 cm^{-1} and 1637 cm^{-1} were attributed to the stretching vibration of hydroxyl groups and carbonyl groups, respectively. Also, all signals shown by the spectrum in Figure 1b (relative intensity, shapes and stretching frequencies) were well preserved compared to those shown by the FT-IR spectrum in Figure 1a. This indicates that there were no considerable changes observed in terms of the chemical structure (functional groups and hydrogen bonding network) of the onion peels recovered from a local waste disposal site compared to onion peels obtained from a local supermarket.

The metal content of the recovered onion peel (0.8 g) was analysed using ICP-OES and resulted in the following outputs: K (22927.00 ± 0.47 ppm), Na (14930.00 ± 0.21 ppm), Mg (7896.00 ± 6.26 ppm), S (2225.00 ± 1.44 ppm), P (2172.00 ± 2.13 ppm), Fe (1815.00 ± 1.03 ppm), Al (1366.00 ± 1.07 ppm), Sr (300.00 ± 2.52 ppm), Mn (216.00 ± 2.18 ppm), B (107.00 ± 0.09 ppm), Ba (53.00 ± 0.23 ppm), Zn (52.00 ± 0.11 ppm), Rb (35.00 ± 0.02 ppm), La (15.00 ± 0.31 ppm), Ga (15.00 ± 0.12 ppm), Cu (13.00 ± 0.17 ppm) and Pb (9.80 ± 0.23 ppm).

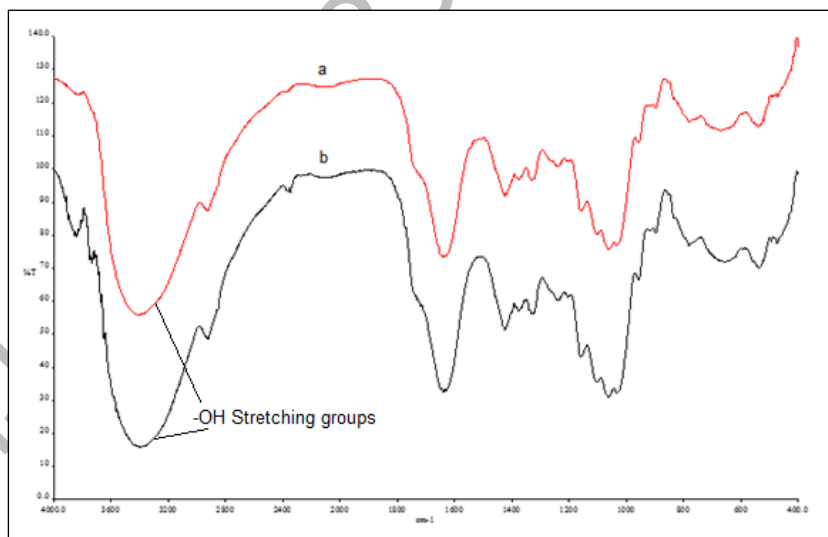


Figure 1. The FTIR spectra of (a) onion peels obtained from a local supermarket and (b) recovered onion peels from a local waste disposal site

For the optimisation of reaction condition, phthalic anhydride **1** and aniline **2** were chosen as the model starting materials. Initially, phthalic anhydride **1** (1.0 mmol) was reacted with aniline **2** (1.0 mmol) in the presence of 2.0 mL of H_2O at $50\text{ }^\circ\text{C}$ to produce the corresponding product, **3a** (Table 1). After 3 hours, the yield of **3a** was found to be poor (Table 1, entry 1). It is worthy to note that when the H_2O was replaced with WEOPA under identical

reaction condition, **3a** was obtained at a moderate yield (54%) (Table 1, entry 2). The amount of WEOPA used was also studied. The usage of 2.0 mL of WEOPA as solvent has produced a good yield of **3a** (91%) (Table 1, entry 7). Interestingly, increases in the amount of WEOPA used did give rise to a better yield of **3a** (Table 1, entry 7). However, pro-longing the reaction time and increasing in the temperature used did not result in a better of yield of **3a** (Table 1, entries 9-10). Based on Table 1, the optimised condition was determined as 2.0 mL of WEOPA, 8 hours and 80 °C (Table 1, entry 7).

Table 1. The optimization of reaction condition for the synthesis of **3a**

Entry	Solvent (mL)	Time (hours)	Temperature (°C)	Yield (%)
1	H ₂ O (2.0)	3	50	30
2	WEOPA (0.5)	3	50	54
3	WEOPA (1.0)	3	50	60
4	WEOPA (2.0)	3	50	69
5	WEOPA (2.0)	3	80	78
6	WEOPA (2.0)	5	80	84
7	WEOPA (2.0)	8	80	91
8	WEOPA (3.0)	8	80	89
9	WEOPA (2.0)	8	100	90
10	WEOPA (2.0)	10	80	89

Next, the study on the general applicability of the WEOPA catalytic system was carried out using different anilines for the synthesis of isoindoline-1,3-dione derivatives under the optimised condition selected above. As shown in Table 2, the isoindoline-1,3-dione derivatives were obtained at moderate to good yields after silica gel column purification (64-91% yield). As indicated in Table 2, the anilines bearing the electron-donating groups (CH₃, OCH₃ and (CH₃)₂CH) afforded the corresponding isoindoline-1,3-dione derivatives **3b-3f** at moderate to good yields (Table 2, entries 2-6) compared to anilines bearing electron-withdrawing groups (F, Cl and Br) (Table 2, entries 7-9). In addition, the current protocol was considered as unprecedented compared to previous methods, because no solvents and additives were required for the synthesis of isoindoline-1,3-dione derivatives. Moreover, the isoindoline-1,3-dione derivatives were synthesized at a reasonable reaction time. The synthesized isoindoline-1,3-dione derivatives were characterised using spectroscopic techniques such as NMR (¹H and ¹³C) and GC-MS analyses.

Table 2. Isolated yields and influence of the WEOPA catalytic system on the synthesis of isoindoline-1,3-dione derivatives

Entry	R ₁	Product	Reaction Time (hours)	Isolated Yield (%)
1	H	3a	8	92
2	2-CH ₃	3b	8	74
3	3-CH ₃	3c	8	70
4	4-CH ₃	3d	8	73
5	4-OCH ₃	3e	8	88
6	4-(CH ₃) ₂ CH	3f	8	75
7	4-F	3g	8	64
8	4-Cl	3h	8	66
9	4-Br	3i	8	68

In addition, we also examined the reusability of the WEOPA catalytic system using the model reaction. After the completion of reaction between phthalic anhydride **1** and aniline **2**, the recovered WEOPA was reused for at least 5 times without any significant loss in catalytic activity (Figure 2).

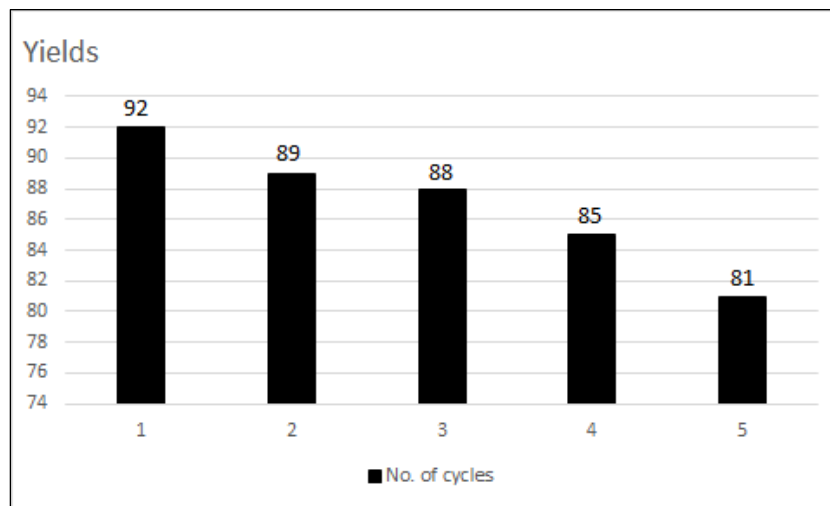


Figure 2. Reactivity of WEOPA based on five runs. Reaction conditions: 1.0 mmol of phthalic anhydride, 1.0 mmol of aniline, 2 mL of WEOP, 80 °C

At the moment, the mechanism of formation of isoindoline-1,3-dione derivatives catalysed by the WEOPA system is not well understood. However, metal content analysis showed that the onion peel ash consists of metal oxides including phosphorus, magnesium, zinc, manganese and sodium as major elements, accompanied by other minor constituents. We hypothesised that the metals in the WEOPA have facilitated the formation of isoindoline-1,3-dione derivatives and the proposed mechanism was shown in Figure 3.

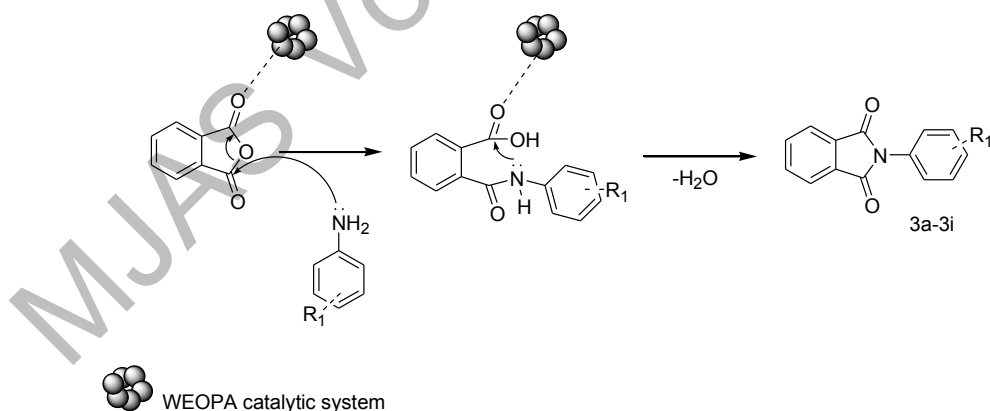


Figure 3. Proposed mechanism for the formation of isoindoline-1,3-dione derivatives catalyzed by the WEOPA

Conclusion

In this work, we have developed a simple, inexpensive and recyclable catalytic system for the synthesis of isoindoline-1,3-dione derivatives. Overall, the yields of the synthesized products were obtained in moderate to good yields (62-91%). In addition, the WEOPA could be recovered and reused for up to five times without any significant

loss in catalytic activity. It is worthy to note that the current protocol can be used to synthesize the isoindoline-1,3-dione derivatives without the presence of any additives, external acids or solvents. The results of this study might induce researchers to perform organic transformations using the WEOPA as a green solvent.

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