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Synthesis of poly(itaconic acid) and its application for synthesis of pyrrolinones as a reusable homogeneous catalyst

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Abstract: In this context, poly(itaconic acid) (PIA) was synthesized through the polymerization of itaconic acid in the presence of $K_2S_2O_8$ and NaH_2PO_4 , achieving satisfactory yields of 80 % within 48 h at 55 °C. The PIA served as a homogeneous catalyst in MeOH for the synthesis of pyrrolin-2-ones from aromatic aldehydes, anilines and dimethyl acetylenedicarboxylate (DMAD). The reactions proceeded efficiently, yielding excellent results with conversion rates of 90–95 % within 8 h at room temperature. The synthesized products were characterized by combustion analysis (CHN), 1H -NMR, ^{13}C -NMR and FT-IR spectra. This method is eco-friendly and aligns with the principles of green chemistry.

Keywords: poly(itaconic acid); pyrrolin-2-ones; green chemistry; homogeneous catalyst.

INTRODUCTION

Polymer catalysts are useful for performing chemical reactions. Polymer catalysts are polymers that possess catalytically active sites. Typically, these active moieties are incorporated into the polymer chains. Examples include the asymmetric Diels–Alder reaction catalyzed by a helical polymer (optically active),¹ the preparation of N-methylated compounds from alcohols and urea from carbamates or CO_2 fixation by copper nanoparticles on triazine–triamine as a porous organic polymer² and epoxidation reactions by $[Cu(H_2btec)(bipy)]$ as a metal–organic polymer catalyst.³ Other examples are the epoxidation of olefins by a one-dimensional polyoxomolybdate polymer,⁴ cascade carbon–carbon bond-forming reactions by CBAP-1(EDA- SO_3H) as a dual-functionalized porous organic polymer⁵ and the acetalization of carbonyl compounds with alcohols by a hierarchical porous organic polymer.⁶ Additionally, examples include a pro-

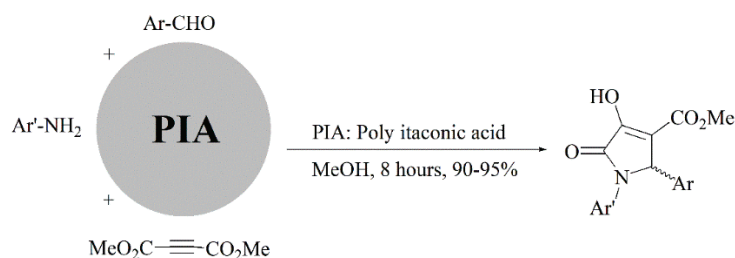
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moted Suzuki reaction using palladium chloride anchored on a polymer,⁷ the synthesis of biomass-derived alkyl levulinates by a sulfonic acid-functionalized organic polymer⁸ and a one-pot multistep organic transformation by a tri-functional covalent triazine and carbonyl-based polymer.⁹ To continue our investigation into the application of DOWEX, a cross-linked copolymer functionalized to act as a heterogeneous catalyst,¹⁰ we have decided to explore the catalytic properties of poly(itaconic acid) (PIA). Because, a comprehensive review of the chemical literature indicates that the catalytic potential of PIA remains unexplored to date. PIA was originally synthesized directly from itaconic acid (IA) by Marvel and Shepherd in 1959.¹¹ The PIA is a major component in some chemical material such as magnetite nanocomposites¹² and hydrogels¹³ (as adsorbent materials), self-assembled clay-polyelectrolyte hydrogels,¹⁴ gelatin-based films (antimicrobial-active food packaging),¹⁵ superabsorbent (a polymer network for application in concrete as an internal curing agent),¹⁶ encapsulator of PCMs (the phase change materials),¹⁷ hydrogels (for human pluripotent stem cell culture),¹⁸ electrospun nanofibers¹⁹ and highly stable fluorescent polymer.²⁰ In this context, PIA was synthesized using a modified method from IA in the presence of $K_2S_2O_8$ and NaH_2PO_4 .

On the other hand, literature surveys indicate that the preparation of pyrrolinone derivatives has been of considerable synthetic interest, driven by the moiety's significant biological activity in numerous natural products and pharmaceutical compounds.²¹

Building on our experience in synthesizing pyrrolin-2-ones,²¹ we evaluated the catalytic properties of PIA through the synthesis of these classes of compounds, as illustrated in Scheme 1.



Scheme 1. The synthesis of pyrrolin-2-ones by poly(itaconic acid) as a homogeneous catalyst in MeOH.

EXPERIMENTAL

All substrates and reagents were obtained from Sigma-Aldrich Company. FT-IR spectra were recorded using a PerkinElmer FT-IR RXI instrument. ¹H- and ¹³C-NMR spectra were obtained on Bruker spectrometers operating at 250, 300 and 400 MHz frequencies. Gel permeation chromatography (GPC) experiments were conducted on an Agilent 1100 series system. The monitoring of reaction progress was performed using Merck silica gel 60 F₂₅₄

aluminum sheets. The mass spectra were recorded using a Agilent 5973N spectrometer at 70 eV. The elemental analysis was performed on the LECO-TruSpec elemental analyzer (CHN, USA). Additionally, ^1H -NMR spectroscopy and combustion analysis were utilized to assess the purity of the synthesized products.

Synthesis of the poly(itaconic acid)(PIA)

A 250 mL flask, equipped with a magnetic stirrer and condenser, was used. An aqueous solution was prepared consisting of itaconic acid (20 g, 153 mmol), sodium dihydrogen phosphate (NaH_2PO_4 , 10 g, 83 mmol), and potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$, 0.2 g, 0.739 mmol) in 100 mL of H_2O . This solution was stirred at a temperature of 55 °C for 48 h. After this, the solution was gradually added to acetone. The PIA was precipitated out of the solution and was then collected through filtration and redissolved in water. This was followed by reprecipitation from acetone, and then the precipitate was dried under reduced pressure. The PIA was obtained as a white powder, with a yield of 80 % or 16 g.

A typical procedure: The synthesis of 1,5-diphenyl-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (1a) in the presence of the PIA as a homogeneous catalyst in MeOH

A mixture of benzaldehyde (0.106 g, 1 mmol), aniline (0.93 g, 1 mmol) and PIA (0.2 g) was prepared in a 25 mL round-bottomed flask using 5 mL of methanol. This mixture was stirred at room temperature for 30 min using a magnetic stirrer. After that, dimethyl acetylenedicarboxylate (DMAD, 0.142 g, 1 mmol) was added to the reaction mixture. The reaction was monitored by thin layer chromatography (TLC) using an ethyl acetate/*n*-hexane solvent system (1:1 ratio) and completed within 8 h. Following the completion of the reaction, the precipitate was filtered and washed with distilled water (3×5 mL). The product was then purified through crystallization from ethanol (which was dried over anhydrous sodium sulfate). The corresponding pyrrolin-2-one was obtained in an excellent yield of 90 % (0.28 g) as a white powder with a melting point range of 182–183 °C (180–182 °C²¹). The product was characterized using by combustion analysis (CHN), ^1H -NMR, ^{13}C -NMR and FT-IR spectra. The corresponding data are given in the Supplementary material to his paper.

Additionally, PIA was recovered from water by the addition of acetone, achieving a yield of 95 % (0.19 g). In this procedure, water was initially evaporated using a laboratory rotary evaporator, leaving behind 12–13 mL of water. This method was employed to minimize acetone usage.

RESULT AND DISCUSSION

Marvel *et al.* synthesized poly(itaconic acid) from itaconic acid using potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$) and hydrochloric acid (HCl, 0.5 M), achieving low yields of 35 %.¹¹ To improve this process, several methods have been published, and the results of these modifications have been summarized in Table I.

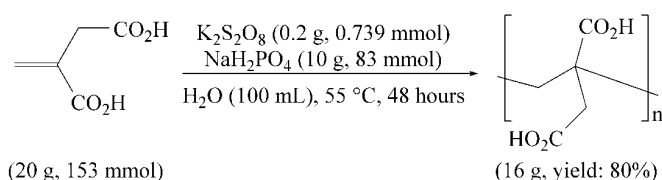
The PIA was synthesized following the protocol outlined by Marvel *et al.* (entry 2, Table I), with modifications (entry 1, Table I) as presented in Scheme 2. The polymerization process was conducted at 55 °C to prevent decarboxylation. The new conditions provide several benefits, such as the ready availability of reagents, safer handling, and increased yield, along with reduced processing times compared to some existing protocols.

The PIA obtained was characterized by an FT-IR, ^1H -NMR and GPC spectra. In the FT-IR spectrum of itaconic acid (Fig. 1), bands appear at 3027

cm^{-1} (attributed to $=\text{C}-\text{H}$ stretching vibration), 1636 cm^{-1} (attributed to $\text{C}=\text{C}$ stretching vibration) and 913 cm^{-1} (attributed to $=\text{C}-\text{H}$ bending vibration). These absorptions were not present in the PIA spectrum, indicating successful synthesis of the polymer. It is important to note that although the acid has the potential to form polyester by creating ester bonds, the stretching band for the $\text{C}=\text{O}$ in the FT-IR spectrum of PIA (Fig. 1B) appeared at 1717 cm^{-1} , which does not align with the expected stretching band for $\text{C}=\text{O}$ esters in the range of $1735\text{--}1750 \text{ cm}^{-1}$. Therefore, it can be concluded that polyester formation does not occur under these conditions.

TABLE I. Synthesis of poly(itaconic acid) (PIA) by different protocols from itaconic acid (IA)

Entry	Reagents	$t / ^\circ\text{C}$	Time, h	Yield / %	Reference
1	IA (77 mmol in 50 mL H_2O), $\text{K}_2\text{S}_2\text{O}_8$ (2.9 % in H_2O), NaH_2PO_4 (9 % in H_2O)	55	48	80	Current work
2	IA (145 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (0.37 mmol), HCl (0.5 M, 50 mL)	50	68	35	11
3	IA (38.5 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (0.92 mmol), $\text{K}_2\text{S}_2\text{O}_5$ (1.1 mmol), HCl (0.1 M, 50 mL)	25	72	70	22,23
4	IA (770 mmol), NaOH (31 g, in 50 mL H_2O), <i>tert</i> -butyl hydroperoxide (0.5 mL, 70 %)	80	1	90	24
5	IA (39 mmol, in 25 mL H_2O), $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (2 mmol), choline chloride (0.91 g)	55	48-96	50-62	25
6	IA (20 mmol), choline chloride (20 mmol), $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (1 mmol in 0.36 ml of distilled water)	110/1 h 65/2 h	3	80	26



Scheme 2. The polymerization of the itaconic acid (IA) to the poly(itaconic acid) (PIA).

The ^1H -NMR spectrum for poly(itaconic acid) was recorded in D_2O . The absence of peaks corresponding to olefinic hydrogens suggests that no monomeric itaconic acid remained within the polymer network, confirming successful polymerization. The other observed peaks in the spectrum were assigned to the appropriate hydrogen groups, as illustrated in Fig. 2.

The polymers were characterized using gel permeation chromatography (GPC). A Waters chromatography system was utilized for the GPC measurements, which employed 0.1 M NaNO_3 as the eluent at a flow rate of 1 mL min^{-1} . The samples were analyzed at a concentration of 1 mg mL^{-1} and a temperature of $23 ^\circ\text{C}$. Calibration was effectively performed using polystyrene standards sup-

plied by Polymer Standards Service. The obtained chromatogram is shown in Fig. 3. The results of the GPC measurements for synthesized poly(itaconic acid) are summarized in Table II.

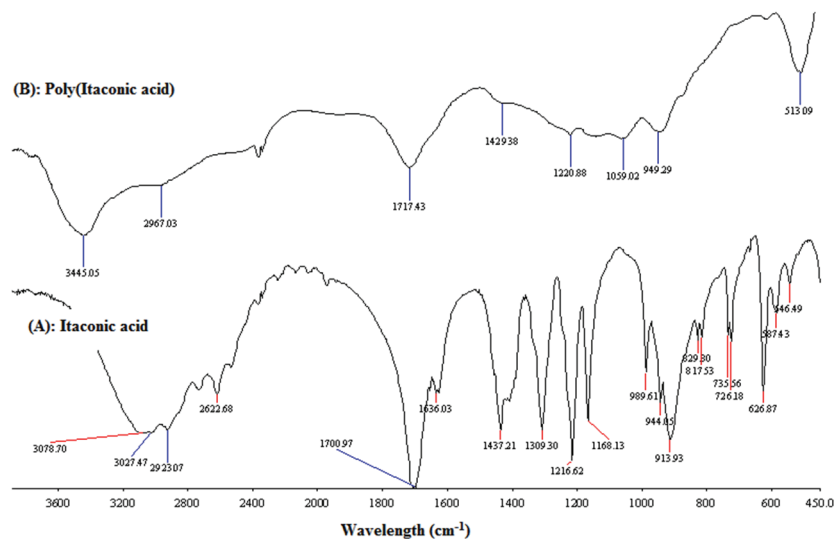


Fig. 1. FT-IR (KBr) spectra of: A) the itaconic acid; B) the poly(itaconic acid).

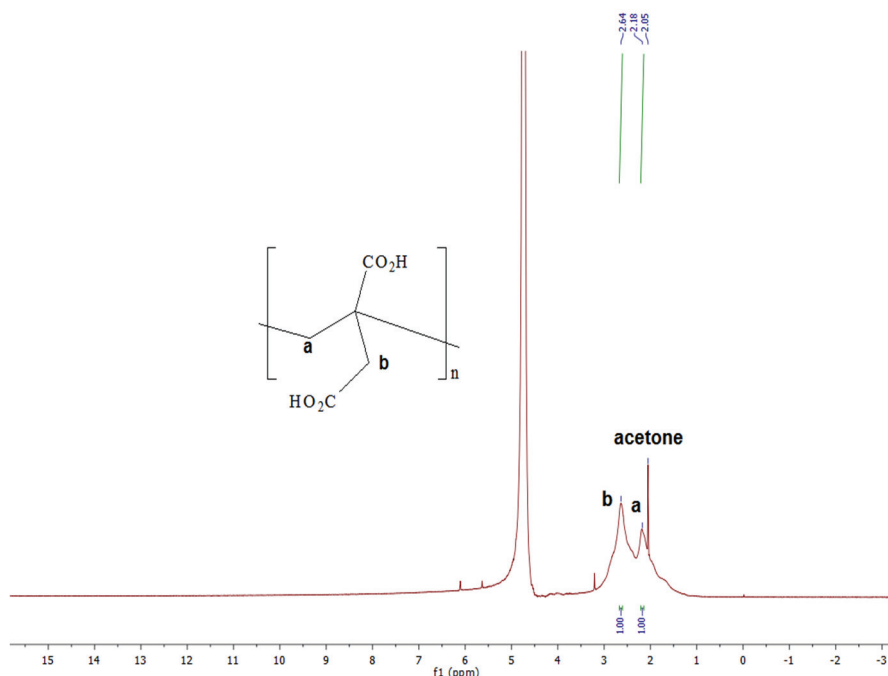


Fig. 2. ¹H-NMR of poly(itaconic acid) sample in D₂O.

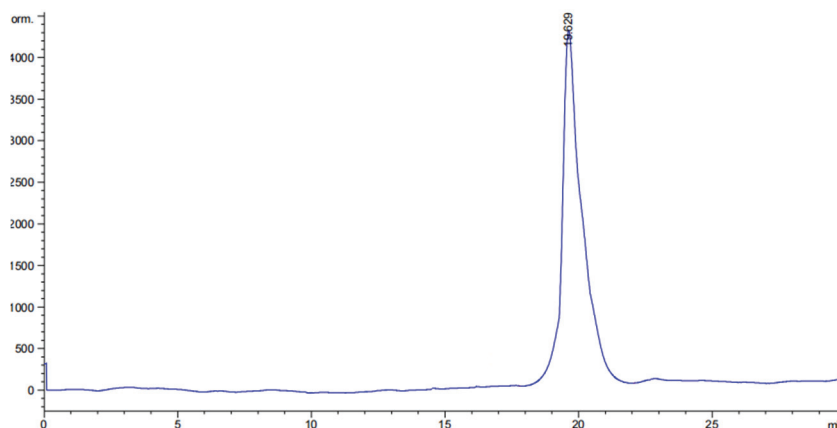


Fig. 3. Gel permeation chromatography (GPC) trace of poly(itaconic acid).

TABLE II. Synthesis parameters and GPC results of poly(itaconic acid) polymerized at 55 °C for 48 h. Z-average molecular weight (M_z), number average molecular weight (M_n), weight average molecular weight (M_w) and polydispersity index (PDI) = M_w/M_n calculated from GPC data

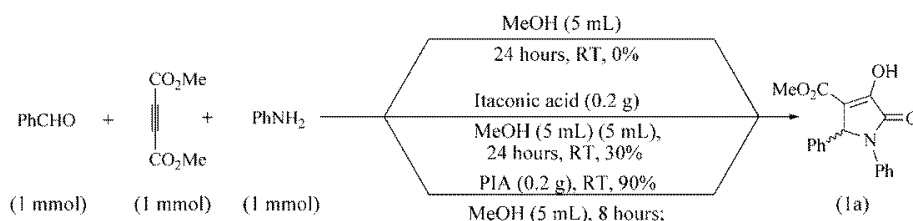
Retention time min	Peak area %	M_z g mol ⁻¹	M_w g mol ⁻¹	M_n g mol ⁻¹	PDI
19.629	100	49251	41654	34879	1.1942

Analysis of the GPC chromatogram data revealed one distinct peak with a retention time of 19.629 min with accounts for a percentage of 100 % total peak area, indicating that it constitutes the major component of the sample. Furthermore, ¹H-NMR and FT-IR spectroscopic analyses have detected no impurities, by-products or additives. Therefore, the chromatographic result is consistent with other analytical methods, ruling out the presence of non-polymer materials.

PIA demonstrates versatile solubility properties, being soluble in methanol and water which makes it a potential candidate for use as a homogeneous catalyst, especially due to the acidic nature (pH 4) of its aqueous solution. This feature allows it to effectively load hydronium ions in methanol-based reactions, creating a moderately acidic environment ideal for moisture-insensitive organic reactions.

A review of the chemistry literature revealed that the catalytic properties of PIA have not been explored to date. The catalytic capabilities of PIA can be explored in a variety of organic reactions. Additionally, this polymer is insoluble in ethanol, propanol and acetone but exhibits solubility in water and methanol.¹¹ Given that pyrrolin-2-ones are frequently synthesized in methanol,²¹ the use of PIA in this synthesis presents a valuable opportunity to evaluate its catalytic efficiency. The synthesis of pyrrolin-2-one derivatives using PIA as a homogeneous catalyst in methanol could provide significant insights into its catalytic properties and potential applications in organic synthesis.

A one-pot and three-component synthesis was carried out for the preparation of 3-acyl-5-hydroxy-3-pyrrolin-2-ones from ArCHO, ArNH₂ and dimethyl acetylenedicarboxylate (DMAD) by PIA as a homogeneous catalyst in MeOH as shown in Scheme 3. The synthesis process of 3-acyl-5-hydroxy-3-pyrrolin-2-ones demonstrates efficiency when PIA is used as a catalyst. Using 0.2 g of PIA has been identified as the optimally efficient quantity for converting benzaldehyde, aniline and dimethyl acetylenedicarboxylate in methanol at room temperature. This highlights the usefulness of PIA in this context. In particular, the derivative 1,5-diphenyl-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (**1a**) achieved high yields of 90 % within 8 h in methanol. Comparing this to the lack of significant yield without a catalyst or with itaconic acid further emphasizes the superior catalytic role of PIA in this synthesis.



Scheme 3. Comparison of the synthesis of 1,5-diphenyl-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (**1a**) without catalyst and by the PIA as a heterogeneous catalyst in ethanol.

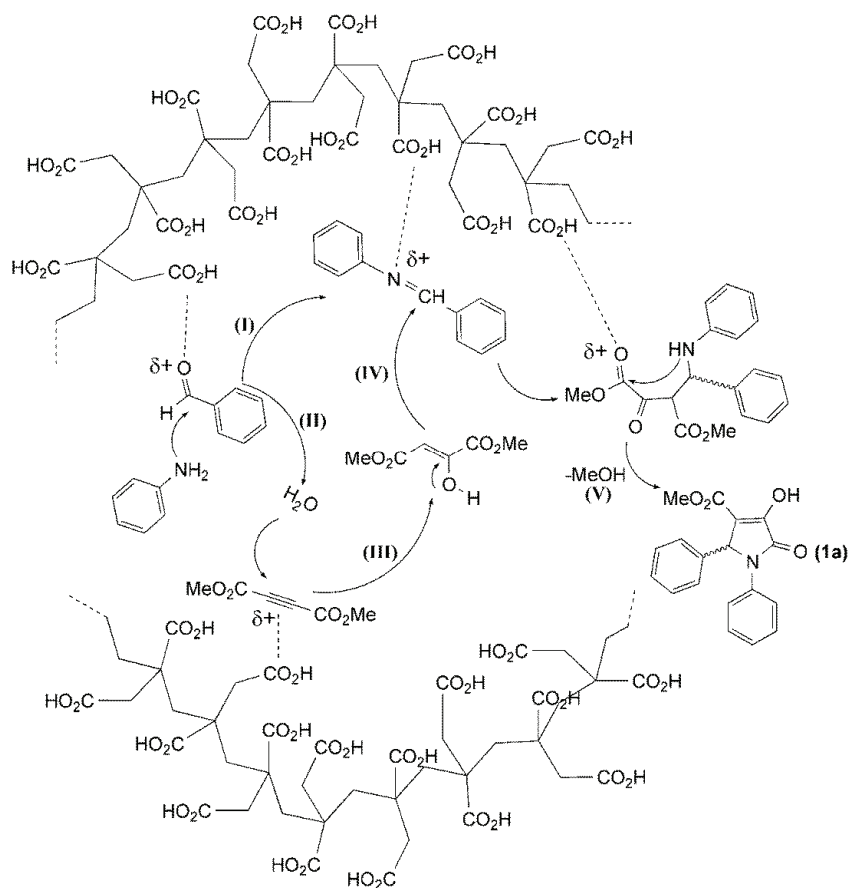
The efficiency of these procedures was evaluated through the synthesis of additional derivatives of 3-acyl-5-hydroxy-3-pyrrolin-2-one under optimized reaction conditions. The corresponding products were synthesized with excellent yields of 90–95 % were achieved in MeOH as indicated in Table III. The products were characterized by CHN, ¹H-NMR, ¹³C-NMR and FT-IR (KBr). Also, the melting point was measured and compared with appropriate references.

TABLE III. The synthesis of pyrrolin-2-ones from Ar-CHO (1 mmol), Ar-NH₂ (1 mmol), DMAD (1 mmol) and the PIA (0.2 g) in MeOH (5 mL) at room temperature; the yields were referred to isolated products. The purity of the products was determined by ¹H-NMR

Entry	Ar-CHO	Ar-NH ₂	m.p. / °C	m.p. (lit.) / °C	Yield / %
1a ^{21,27,28}	Ph	Ph	182–183	180–182	90
2a ^{21,27}	4-Br-Ph	4-Br-Ph	217–219	218–220	92
3a ³⁰	Ph	4-MeO-Ph	171–173	170–171	90
4a ²¹	2-Cl-Ph	4-MeO-Ph	191–193	192–194	90
5a ²¹	4-Br-Ph	4-Me-Ph	170–172	170–172	95
6a ²¹	2-Cl-Ph	4-Me-Ph	192–194	193–194	90
7a ²¹	3-Br-Ph	4-MeO-Ph	170–171	170–171	94

As illustrated in Scheme 4, the synthesis of pyrrolin-2-ones involves a multi-step process, where PIA plays a crucial role in activating and transforming the

reactants. The first step involves the acid surface of PIA activating the aldehyde functional group, enabling it to react with aniline and form an imine intermediate. In the subsequent condensation reaction, water molecules are released and react with dimethyl acetylenedicarboxylate, generating a 1,3-dipolar intermediate.²⁹ This intermediate then adds to the activated imine. The following reaction involves the amino group attacking one of the ester groups through an intramolecular process. The reaction proceeds through five-membered ring formation, establishing the pyrrolin-2-one system. Notably, the enol form represents the more stable configuration in these compounds. It is noteworthy that PIA facilitates this step of the reaction, further highlighting its catalytic proficiency in the synthesis of pyrrolin-2-ones. The PIA, once recovered, can be used in subsequent reaction cycles without any limitations. Despite this, there was no observation of leakage from the catalyst or its components in the products.



Scheme 4. The proposed mechanism for the synthesis of pyrrolin-2-one derivatives by the PIA.

Comparisons between these protocols and other reported methods for synthesizing pyrrolin-2-one derivatives are presented in Table IV. Based on the results obtained, the PIA catalyst exhibits a favorable profile for the synthesis of the pyrrolinone skeleton. The higher product yields, shorter reaction times, and the polymer's recyclability, compared to some existing methods, represent its key catalytic advantages.

TABLE IV. Comparison of synthesis of product **1a** by different methods in the literature²¹

Entry	Protocol	Yield / %	Time	Reusable catalyst	Reference
1	PIA in MeOH	90	8 h	Yes	Current
2	Fe ₃ O ₄ @PEG-400@OA	94	24 h	Yes	21a
3	AmberChrom/EtOH	90	24 h	Yes	21b
4	Fe ₃ O ₄ @NH ₂ @OA	95	8 h	Yes	21c
5	TiO ₂ admicelle/H ₂ O	75	18 h	Non	27
6	Fe ₃ O ₄ @SiO ₂ @propyl-ANDSA/EtOH	87	8 h	Yes	28
7	P-TsOH/EtOH	62	24 h	Non	29
8	EtOH/H ₂ O	87	15 h	Non	30
9	Citric acid/US/EtOH	90	20 min	Non	31
10	P-TsOH/MW/EtOH	96	6 min	No	32
11	[bmim]BF ₄ /PEG-400	89	1 h	Yes	33
12	MW/[BBSI]Cl/glycol	90	5 min	Yes	34
13	[BBSI]Cl/ball milling	90	25 min	Yes	35

CONCLUSION

In this study, poly(itaconic acid) (PIA) was synthesized using potassium persulfate and sodium dihydrogen phosphate with a good yield of 80 % within 48 h at 55 °C. Notably, this represents the first reported application of PIA as a homogeneous catalyst. When employed in methanol for pyrrolin-2-one synthesis, PIA demonstrated satisfactory catalytic performance. Compared to some existing methods, this polymer offers significant catalytic advantages, such as higher product yields, shorter reaction times and recyclability. Consequently, PIA can be introduced as a convenient and reusable catalyst for conducting organic reactions with a green profile. Future research could be directed toward examining the catalytic properties of PIA derivatives, such as novel co-polymers, for executing organic reactions.

SUPPLEMENTARY MATERIAL

Additional data and information are available electronically at the pages of journal website: <https://www.shd-pub.org.rs/index.php/JSCS/article/view/13026>, or from the corresponding author on request.

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ИЗВОД

СИНТЕЗА ПОЛИ(ИТАКОНСКЕ КИСЕЛИНЕ) И ЊЕНА ПРИМЕНА КАО ВЕШЕКАРНОГ
ХОМОГЕНОГ КАТАЛИЗАТОРА ЗА СИНТЕЗУ ПИРОЛИНОНА

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Поли(итаконска киселина) (PIA) је синтетисана полимеризацијом итаконске киселине у присуству $K_2S_2O_8$ и NaH_2PO_4 , у добром приносу од 80 %, током 48 h, на температури од 55 °C. PIA је коришћена као хомогени катализатор за синтезу пирулин-2-она из ароматичних алдехида, анилина и диметил-ацетилендикарбоксилата (DMAD). Реакција се врши у метанолу, у кратком реакционом времену, у одличном приносу од 90–95 %, током 8 h на собној температури. Синтетисани производи окракатреисани су микроанализом (CHN), 1H -NMR, ^{13}C -NMR и FT-IR спектрима. Описана метода је еколошки прихватљива и у складу је са принципима зелене хемије.

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