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Ultra-Sonicated Synthesis Of 1,8-Dioxo-Octahydroxanthenes Ni Multi-Doped Zro$_2$ Nanocatalyst

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ABSTRACT

In this study, we report the efficient synthesis of 1,8-dioxo-octahydroxanthenes in excellent yields via the condensation of aldehydes with dimedone, catalyzed by Ni multi-doped ZrO_2 in aqueous medium under both ultrasonic and conventional conditions. This catalytic system offers several advantages, including high yields, strong catalytic activity, operational simplicity, non-toxicity, and an easy work-up procedure. Furthermore, the nanocatalyst can be readily recovered and reused for up to five consecutive cycles without any significant loss of activity.

Keywords: *ultra-sonication, nanocomposite, heterocyclic compounds.*

INTRODUCTIONS

Among the various nitrogen- and oxygen-containing heterocyclic compounds, xanthene derivatives hold remarkable structural and functional importance. Heterocyclic frameworks constitute a fundamental class of organic molecules and play a vital role across diverse scientific and technological domains, including agriculture, industrial chemistry, pharmaceuticals, medicinal chemistry, and advanced materials science. [1,2] Within this broad category, xanthenes represent a particularly significant class of heterocycles due to their wide spectrum of biological and pharmacological activities. Numerous reports have highlighted their antiviral, anti-inflammatory,[3] anticancer,[4] antibacterial,[5] antifungal, anti-tuberculosis,[6] and anti-diabetic properties, as well as their ability to interact with DNA.[7] These attributes make xanthene scaffolds valuable targets in drug discovery and medicinal chemistry.

In recent years, considerable effort has been devoted to the development of efficient catalytic systems for the synthesis of 1,8-dioxo-octahydroxanthene derivatives, often through the condensation of dimedone with aryl aldehydes. Notably, various transition-metal, have been employed in this transformation, leading to improved yields and reaction efficiencies. [8,9] As is well recognized, catalysts play a pivotal role in enhancing the rate, selectivity, and sustainability of chemical reactions, and are thus considered one of the foundational pillars of modern chemical science. [10,11]

The increasing demand for environmentally benign and cost-effective processes has intensified the search for green and sustainable catalysts. In this context, nanocatalysts have emerged as a rapidly advancing and influential area in catalytic chemistry, owing to their high surface area, tunable physicochemical properties, enhanced activity, and potential for recyclability. These features position nanostructured catalytic materials as promising candidates for addressing current challenges in sustainable chemical synthesis.

Sonochemistry uses ultrasonic vibrations to accelerate chemical reactions and is widely employed in organic synthesis due to its efficiency and simplicity. Ultrasound spans 20 kHz to 10 MHz and is classified into low-, medium-, and high-frequency ranges. Ultrasonic irradiation often enhances yields and shortens reaction times, making sonochemistry a valuable method for the rapid synthesis of heterocyclic and fused heterocyclic compounds. In this context, we investigate the use of Ni multi-doped ZrO_2 as an efficient heterogeneous nanocatalyst for the synthesis of 1,8-dioxo-octahydroxanthenes under ultra sonicator, employing ethanol as a green solvent. This study underscores the potential of multi-doped nanocatalysts in promoting sustainable heterocyclic synthesis.

EXPERIMENTS

MATERIALS AND METHODOLOGIES

The chemicals used in this study—including aryl aldehydes, dimedone, ethyl acetate, petroleum ether, and ethanol—were obtained from Merck, Aldrich, Fluka, and Loba. Melting points were measured in open capillary tubes and are reported without correction. Reaction progress was monitored by thin-layer chromatography (TLC) using silica gel 60 F254 aluminum plates (0.25 mm thickness, Merck). Sonicator is used for the synthesis of substituted 1, 8-Dioxo-octahydroxanthenes (Q125 Sonicator Ultrasonic Homogenizer). The synthesized products were characterized by comparing their spectral (IR, ^{1}H NMR, ^{1*}C NMR) and physical data with those reported in the literature. NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer.

Preparation of catalyst system

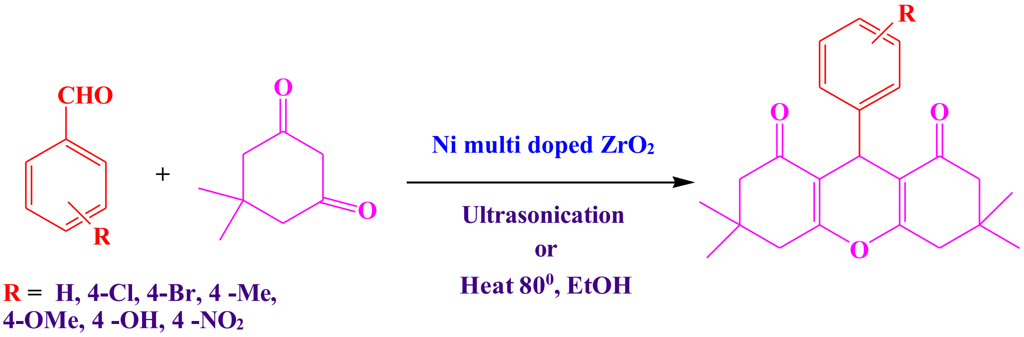
The Ni, C, N, and S multi-doped ZrO_2 catalyst (NiZr) was prepared by dissolving $ZrO(NO_3)_2$ in dilute HNO_3, followed by the controlled addition of PEG-200. After stirring, an appropriate amount of $Ni(NO_3)_2{\cdot}6H_2O$ was introduced to achieve Ni:Zr ratios of 0.3%, 0.6%, or 1.0%. Thiourea was then added, and the mixture was stirred until a uniform solution formed. The resulting precursor was dried at 100 °C and subsequently calcined at 500 °C to obtain the doped ZrO_2 powder. The catalyst was characterized using XRD, TEM, SEM, EDS, FT-IR, and UV-DRS techniques [12].

Conventional method for the synthesis of substituted 1, 8-Dioxo-octahydroxanthenes.

Ni multi-doped ZrO_2 nanocatalyst (0.08 g) was added to a round-bottom flask containing aryl aldehyde (0.5 mmol) and dimedone (1 mmol) in ethanol, and the mixture was heated at 70–80 °C under reflux for the time specified in Table 1 (Scheme 1). Reaction progress was monitored by TLC. After completion of the reaction, the catalyst was recovered by filtration and washed with ethanol. The solvent was removed under reduced pressure to obtained desired product. The product was dried and recrystallized from suitable solvent. The catalyst demonstrated excellent recyclability, showing no significant loss in yield over five consecutive cycles.

Ultra-sonication method for the synthesis of substituted 1, 8-Dioxo-octahydroxanthenes

In a beaker, aryl aldehyde (0.5 mmol) and dimedone (1 mmol) were reacted in ethanol using a Ni multi-doped ZrO_2 nanocatalyst was subjected to ultrasonic waves for the indicated period of time at room temperature to obtain 1,8-dioxo-octahydroxanthenes (Scheme 1). The reaction mixture was irradiated with a time interval of 1 min and the reaction progress was monitored using TLC. After completion, the mixture was quenched with 20 mL distilled water, and the resulting solid was washed and air-dried. The product is dissolve in ethyl acetate and the catalyst was removed by filtration. The organic layer was evaporated, and the crude product was dried and recrystallized from ethanol. The catalyst maintained its activity over multiple cycles.



Scheme 1. Synthesis of 1, 8- Dioxo- octahydroxanthene derivatives under conventional and ultra-sonication method

Optimization of the multi-doped Ni–ZrO_2 nanocatalyst loading

Substituted aryl 1,8-dioxo-octahydroxanthene derivatives were synthesized from aryl aldehyde (0.5 mmol) and dimedone (1 mmol) using a Ni multi-doped ZrO_2 nanocatalyst under ultrasonic irradiation in ethanol at room temperature, as well as by a conventional heating method at 80 °C. As shown in Table 1, 0.08 g of the catalyst was sufficient to afford products of high purity under both conditions. Increasing the catalyst amount did not significantly improve the yield while only a trace amount of product was formed in the absence of the catalyst.

Entry	**Catalyst (g)**	**Ultra-sonication method**		**Conventional method**	
		Time	**Yield %**	**Time**	**Yield %**
1.	0.02	50	87	60	80

2.	0.04	45	88	55	84
3.	0.06	43	92	50	88
4.	**0.08**	**35**	**96**	**45**	**92**
5.	0.10	35	94	50	91
6.	0.12	40	90	30	88
7.	0.14	40	90	60	85
8.	None	90	trace	60	trace

Table 1: Effects of the amount of catalyst on ultra-sonication and conventional methods.

Result and conclusions:

The synthesis of 1,8-dioxo-octahydroxanthenes derivatives was carried out using a Ni multi-doped ZrO_2 nanocatalyst under two different reaction setups, namely conventional heating and ultrasonication methods. A series of aromatic aldehydes were condensed with dimedone under the optimized reaction conditions to investigate the scope and applicability of the present methodology. The progress and completion of all reactions were monitored by thin-layer chromatography, and the reaction time as well as product yield were recorded and shown in Table 2. The dimedone and substituted aldehydes undergoes cyclization in the presence of a small amount of catalyst within a short reaction time, furnishing products in moderate to excellent yields. The results shown in Table 2 (entry 1) under optimized conditions clearly demonstrate the reactivity of aryl aldehydes with dimedone in the presence of the catalyst, yielding xanthene derivatives. The results showed that aldehydes with electron-withdrawing groups exhibited greater efficiency than those with electron-donating groups (see in Table 2). Electron-withdrawing substituents shortened the reaction time and improved the overall efficiency of the reaction. The synthesis of the target pure 1,8-dioxo-octahydroxanthenes derivatives was successfully achieved, and their structures were confirmed using of various analysis technique such as IR, NMR, and MS spectroscopy.

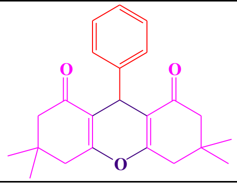
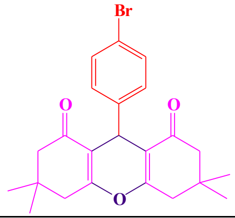
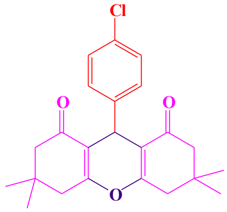
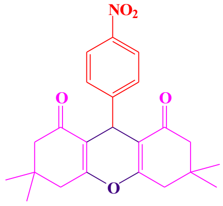
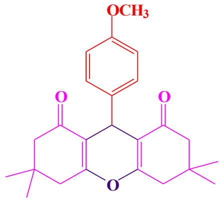
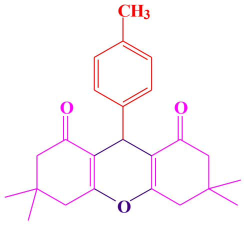
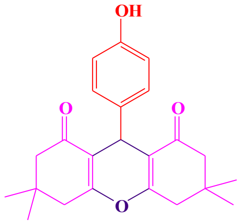
The synthesis of 1,8-dioxo-octahydroxanthene derivatives was performed using a Ni multi-doped ZrO_2 nanocatalyst under two different reaction setups, namely conventional heating and ultrasonication methods. A variety of aromatic aldehydes were reacted with dimedone under the optimized catalytic conditions to evaluate the substrate scope and efficiency of the methodology. Reaction progress was monitored by thin-layer chromatography, and the corresponding yields and reaction times are summarized in Table 2.

In both methods, dimedone and substituted aldehydes goes smooth cyclization in the presence of a small amount of catalyst, delivering the desired xanthenes in moderate to excellent yields within short reaction times. The data in Table 2 clearly highlight the effectiveness of the optimized conditions, demonstrating the catalytic reactivity of Ni multi-doped ZrO_2 nanocatalyst toward promoting the formation of 1,8-dioxo-octahydroxanthene derivatives.

A noteworthy trend was observed with respect to the electronic nature of the aldehyde substituents. Aldehydes bearing electron-withdrawing groups showed enhanced reactivity, resulting in shorter reaction times and higher yields compared to those with electron-donating substituents. This improvement can be attributed to the increased electrophilicity of the carbonyl group, which facilitates the Knoevenagel condensation followed by intramolecular cyclization. Overall, the methodology proved to be efficient, versatile, and reproducible. The final products were obtained in high purity, and their structures were fully confirmed using spectroscopic techniques including IR, NMR, and MS analyses.

Table 2. Synthesis of 1,8-dioxo-octahydroxanthenes using Ni multi-doped ZrO_2 nanocatalyst

	R	**Product**				**Melting point**

Sr. No .			**Time (min)**	**Yield (%)**	**Melting point (°C)**	**reported (ref)**
1.	C_6H_5		50	90	202-204	203-205[13]
2.	4-Br-C_6H_4		40	91	238-240	238–240[14]
3.	4-Cl-C_6H_4		38	92	236-238	234-236[15]
4.	4-NO_2-C_6H_4		30	96	228-232	230-232[16]
5.	4-OMe-C_6H_4		50	87	240-242	237-240[17]
6.	4-Me-C_6H_4		45	88	217-218	215-217 [18]
7.	4-OH-C_6H_4		43	90	250-252	248-250[19]

To clearly demonstrate the advantages and improved efficiency of the present method compared to previously reported approaches for synthesizing 1,8-dioxo-octahydroxanthene derivatives, key comparative parameters such as yield, reaction time, and catalyst performance are summarized in Table 3. A detailed examination of these data shows that the Ni multi-doped ZrO_2 nanocatalyst offers significant benefits, most notably higher product yields and substantially shorter reaction times. Table 3 highlights the superior performance of the Ni multi-doped ZrO_2 nanocatalyst relative to other catalysts reported in the literature. The present methodology stands out due to its environmentally benign reaction medium, excellent product yields, and efficient catalytic activity under mild conditions. These features collectively make the protocol more sustainable and practical than existing methods.

One of the most important advantages of this system is the excellent recyclability of the catalyst. In the present study, the catalyst was recovered by simple filtration and reused in subsequent reactions with fresh substrates. Remarkably, for the model reaction between benzaldehyde and dimedone, the catalyst retained high activity even after five consecutive cycles, with yields decreasing only slightly from 96% to 90%. This high level of stability and reusability underscores the robustness and economic viability of the Ni multi-doped ZrO_2 nanocatalyst for the synthesis of 1,8-dioxo-octahydroxanthene derivatives.

Table 3. Comparison of the performance of different catalysts in synthesis of the 1,8-dioxo-octahydroxanthene.

Sr. No.	Catalyst	Solvent	Energy Medium	Temperature (ºC)	Time, (min)	Yield (%)	Ref.
1.	Fe_3O_4@THAM-Piperazine (2020)	H_2O:EtOH	heat	100	30	84	[20]
2.	$ZnFe_2O_4$@Fe_3O_4(2023)	EtOH	heat	eflux	45	94	[21]
3.	$CuFe_2O_4$@SO_3H(2019)	EtOH	heat	rt	60	95	[22]
4.	$NiFe_2O_4$@Cu)(H^+-Mont) MNPs(2019)	H_2O:EtOH	heat	90	30	93	[23]
5.	MSrGO NCs(2019)	SF	heat	80	60	94	[24]
6.	Au/NiAlTi LDH(2020)	EtOH	heat	reflux	30	90	[25]
7.	Fe_3O_4@OMWCNT(2023)	EtOH	heat	reflux	90	93	[26]
8.	**Ni multi-doped ZrO_2**	**EtOH**	**U.S**	**rt**	**30**	**96**	**This work**
			heat	80	45	92	

CONCLUSION

In this study, 1,8-dioxo-octahydroxanthenes were efficiently synthesized from aromatic aldehydes and dimedone using a Ni multi-doped ZrO_2 nanocatalyst. An ultrasonication-assisted approach was employed, enabling the synthesis of various derivatives at room temperature in ethanol. This method offers several notable advantages, including simple product isolation and purification, excellent yields, and the effective recyclability of the Ni multi-doped ZrO_2 nanocatalyst for up to five cycles with no significant loss in catalytic performance.

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CHARACTERIZATION DATA :-

1) 3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-phenyl-2H-xanthene-1,8(5H,9H)-dione

White colour; m.p. 2002-204 ºC; **^{1}H NMR, (CDCl3), δ (ppm);** 7.29 (m, 2H), 7.21(dd,2H),7.10 (m, H) 4.75 (S, 1H), 2.47(s, 4H), 2.19 (m, 4H),1.10 (S, 6 H), 0.99 (S, 6 H). **^{13}CNMR (CDCl3) δ (ppm):** 27.34,29.29,31.84,32.03,32.21,40.87,50.76,115.66,126.37,128.05,128.39,144.12,162.29,196.40.

FT-IR (KBr) cm^{-1}: 2955.71, 2870.11, 1658.04, 1623.29, 1490.98, 1466.01, 1234.77, 1162.25, 1137.17, 1019.69, 999.87, 932.16, 912.24, 891.14, 841.21, 797.96, 741.89, 697.03, 655.29, 605.61, 570.33, 553.85, 522.24.

HRMS (ESI) $C_{23}H_{27}O_3$: [M+H] Analysis and calcd for 351.1962.

2) 9-(4-bromophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione

White Colour; m.p. 238-240 °C; **^{1}H NMR (CDCl3), δ (ppm);** 7.33 (m, 2H), 7.18(m, 2H),4.6 (S, 1H), 2.47 (m, 4 H), 2.14-2.25 (q, 4 H), 1.10 (S, 6 H),0.98 (S, 6 H). **^{13}CNMR (CDCl3) δ (ppm):** 27.30, 29.31, 31.59, 32.22, 40.82, 50.70, 115.13, 120.21, 130.21, 131.13, 143.30, 162.54, 196.40. **FT-IR (KBr) cm-1:** 2951.10, 2873.65, 657.72 1623.13 1485.86 1467.35 1408.66 1234.78 1163.04 1137.17 1070.14 1003.49 932.32 890.75 847.78 816.57 778.67 710.66 660.71 630.85 605.56 562.83 524.64. **HRMS (ESI)** $C_{23}H_{26}BrO_3$: [M+H] Analysis and calcd for found 431.1047.

3) 9-(4-chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione

White Colour; m.p. 236-238 °C; **^{1}H NMR (CDCl3), δ (ppm);** 7.20 (m, 4H), 4.71 (S, 1H), 2.48 (d, 4H), 2.19 (qd, 4H), 1.10 (S, 6 H), 0.98 (S, 6 H). **^{13}CNMR (CDCl3) δ (ppm):** 27.27, 29.28, 31.26, 31.49, 31.70, 32.01, 32.19, 40.79, 50.70, 115.16, 115.19, 128.17, 129.82, 131.92, 131.94, 142.80, 142.83, 162.55, 162.57, and 196.39. **FT-IR (KBr) cm^{-1}:** 2950.67 2872.90, 1658.42 1624.03 1488.51 1467.96 1412.41 1234.99 1163.33 1137.97 1087.45 1001.78 932.77 890.59 848.49 817.07 780.60 712.32 660.44 634.75 606.13 564.38 526.28. **HRMS (ESI)** $C_{23}H_{26}ClO_3$: [M+H] Anal. Calculated for 385.1567

4) 3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-(4-nitrophenyl)-2H-xanthene-1,8(5H,9H)-dione

White Colour; m.p. 220-222 °C; **^{1}H NMR (CDCl3), δ (ppm);** 8.9 (m, 2H), 7.48 (m,2H),4.83 (s, 1H), 2.51 (m, 4H), 2.26(d, 2H), 2.16 (d, 2H), 1.12 (s, 6H), 0.99 (s,6H).**^{13}CNMR (CDCl3) δ (ppm):**27.28, 29.24, 32.06, 32.24, 32.39, 40.84, 50.61,114.52, 123.42, 129.38, 146.48, 151.57,162.99, 196.29. **FT-IR (KBr) cm-^{1}:** 2957.64 2868.88 1615.00 1512.77 1468.18 1389.43 1359.92 1341.14 1165.12 1137.57 1109.67 1000.80 981.26 891.46 866.50 831.79 740.91 695.19 665.18 631.31 610.57 563.61 520.40. **HRMS (ESI)** $C_{23}H_{26}NO_5$: Anal. calcd for 396.1819.

5) 3,4,6,7-tetrahydro-9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione

White Colour; m.p. 240-242°C; **^{1}H NMR (CDCl3), δ (ppm);** 7.20 (m, 2H), 4.70 (s,1H),3.72 (d, 3H), 2.46 (m, 4H), 2.21(m, 4H), , 1.09 (s, 6H), 0.99 (s, 6H). **^{13}CNMR (CDCl3)δ (ppm):** 27.35, 29.29, 30.97, 32.20, 40.87, 50.79, 55.11, 113.47, 115.79, 129.32, 136.52, 157.96, 162.08,196.48. **FT-IR (KBr) cm-1:** 2958.20 2933.22 2875.24 1661.29 1625.09 1606.85 1509.53 1460.51 1300.76 1258.43 1233.03 1191.18 1163.04 1135.45 1108.37 1031.33 997.21 930.82 910.60 840.85 811.40 774.13 720.67 683.19 631.49 609.25 569.75 555.76 529.79. **HRMS** (ESI) $C_{24}H_{29}O_4$: [M+H]. Anal. calcd for 381.2061.

6) 3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-p-tolyl-2H-xanthene-1,8(5H,9H)-dione

White Colour; m.p. 214-217°C; **^{1}H NMR (CDCl3) ,δ(ppm);** 7.17 (m, 2H), 7.1 (d, 2H),4.71 (S, 1H), 2.46 (s, 4H), 2.20 (m, 8H), 1.66-1.72(m, 4 H), 1.09 (S, 6 H), 0.99 (S, 6H). **^{13}CNMR (CDCl3) δ (ppm):** 21.08, 27.38, 29.28, 31.45, 32.21, 40.88, 50.7, 115.76, 128.26, 128.79, 135.75, 141.24, 162.15, 196.44. **FT-IR (KBr) cm^{-1}:** 2958.24 2926.13 1659.45 1621.89 1510.55 1464.69 1163.71 1135.13 1107.22 1019.05 997.70 930.53 909.76 885.25 840.41 821.77 773.00 715.94 684.28 652.96 629.36 609.47 570.29 555.03 518.84. **HRMS (ESI)** $C_{24}H_{29}O_3$: [M +H]. Anal. calcd for 365.2121.

7) 3,4,6,7-tetrahydro-9-(4-hydroxyphenyl)-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione

White Colour; m.p. 248-250 °C; **^{1}H NMR (CDCl3), δ (ppm);** 7.6 (m, 2H), 6.53(m, 2H), 4.46 (s, 1H), 2.46 (s, 4H), 2.21(m, 4H), 1.09 (s, 6H), 0.99 (s, 6H). **^{13}CNMR (CDCl3) δ(ppm):** 27.42, 29.17, 30.97, 32.28, 40.86, 50.78, 115.31, 115.91, 129.33, 135.46, 154.81, 162.49, 197.34. **FT-IR (KBr) cm^{-1}:** 3382.80 2961.55 2930.83 1679.09 1660.07 1613.83 1593.56 1511.65 1447.66 1245.16 1164.86 1150.88 1132.19 1105.50 1002.84 930.98 888.84 837.40 685.34 628.72 569.76 529.38 507.28. **HRMS (ESI)** $C_{23}H_{27}O_4$: [M +H]. Anal. calcd for 367.1902.

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