

# Modified Dowex-50 W-Promoted Synthesis of Chalcones Containing Hydroxyl and Nitro Groups

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## Abstract

Dowex-50W-sodium ion exchange was used after acidification and modification via nitration and sulfonation as a reusable catalyst for the synthesis of chalcones containing hydroxyl and nitro groups through condensation of substituted aldehydes and substituted acetophenone. This synthesis provides several advantages such as excellent yields of the required products. The prepared compounds (1-11) were identified by m.p., IR, CHN analysis, <sup>1</sup>H, and <sup>13</sup>C-NMR.

**Keywords:** Dowex resin, chalcones, Claisen-Schmidt condensation

## 1. Introduction

Chalcones have been used as intermediates for the preparation of compounds having therapeutic value. Literature review revealed that chalcone derivatives exhibited diverse pharmacological activities; such as potential cytotoxic agents, antimicrobial agents, antiviral and anti-inflammatory [1-4]. Chalcones can also be used as precursors for the synthesis of more complex molecules as well as in the synthesis of 1,3-diarypropanol for the study of charge transfer in the field of photochemistry [5]. Based on the above observations it is worthwhile to use different methods to prepare these compounds in a high yield. These compounds are prepared by condensing aryl ketones with aromatic aldehydes in the presence of suitable condensing agent. Basic medium can be used in most cases. However, the acidic medium is required when it is desired to prepare chalcones bearing –OH and –NO<sub>2</sub> groups. With respect to the –OH group, the preparation of chalcones containing –OH (in the aldehyde part) in the basic medium cannot be used since the aldehydic phenol in this medium is not reactive enough due to the strong electron release of the phenoxide anion, which will reduce the electrophilicity of the carbonyl carbon, making the attack on the carbanion (derived from the aromatic ketone) less favourable [6]. Some bromonaphthyl chalcones containing –NO<sub>2</sub> group (in the three position of the aldehyde ring) have been prepared using SiO<sub>2</sub>–H<sub>2</sub>SO<sub>4</sub> as a re-usable catalyst [7] and therefore it was decided to use re-usable Dowex resins for the same purpose. Dowex has been used as a catalyst in the synthesis of oxazoline, imidazoline, thiazoline derivatives [8] and also in the conversion of dicarboxylic acid into monoesters [9].

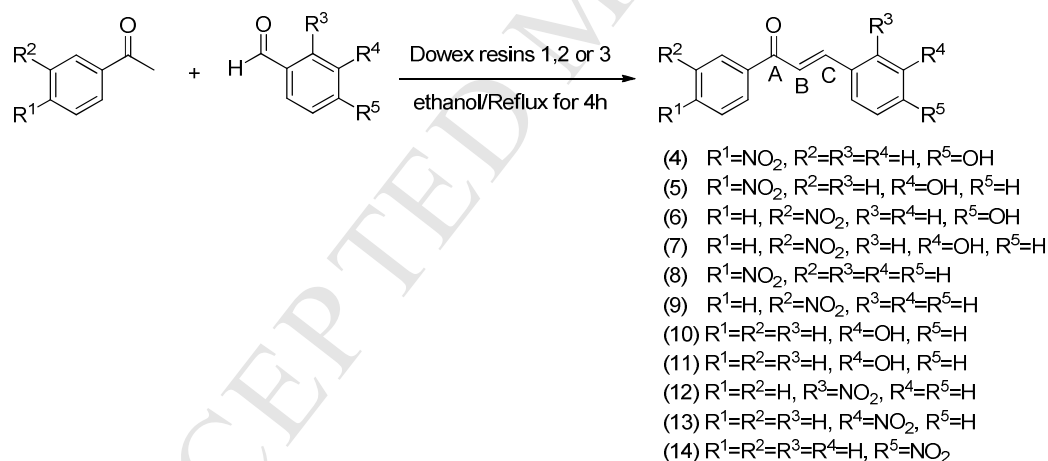
## 2. Results and Discussion

### 2.1. Modification of Dowex Resins:

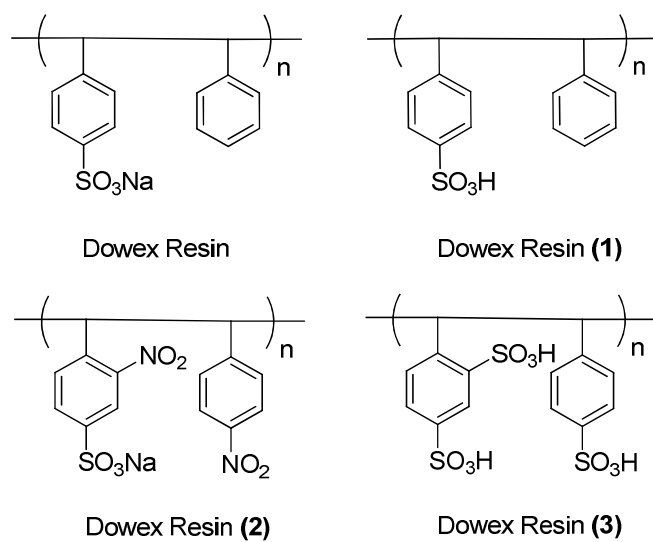
The presence of the functional group in the resin can be identified by IR spectroscopy only due to the insolubility of the resins. The IR spectra of the Dowex in the salt and in the acid forms are almost the same except for the pronounced -OH broad band from the -SO<sub>3</sub>H in the acid form. The nitrated resin showed distinguished -NO<sub>2</sub> absorptions at 1533 cm<sup>-1</sup> (asymmetric stretching) and at (1346 cm<sup>-1</sup>) (symmetric stretching) while -SO<sub>2</sub> group appeared at 1332 cm<sup>-1</sup> (symmetric stretching). All three modified resins 1, 2 and 3 were titrated with NaOH and the results showed that the acidity followed the order 3>2>1.

## 2.2. Preparation of Chalcones:

The preparation of chalcones were carried out using Claisen-Schmidt condensation methodology using equimolar amounts of aldehyde and ketone in the presence of the acidic modified Dowex resins as catalyst and absolute ethanol as solvent. It was found that the yield of some of these chalcones increased by using resin (3) which has the highest percent of -SO<sub>3</sub>H compared with previous studies in the literature [11,12]. The IR spectra of the prepared chalcones showed strong bands in the range (1553-1610 cm<sup>-1</sup>) due to the olefinic C=C stretching and (1643-1666 cm<sup>-1</sup>) due to the conjugated C=O with the double bond which appeared at lower range due to the conjugation [13]. Also the spectra showed bands in the range 1447-1582 cm<sup>-1</sup> due to the aromatic double bond, while the phenolic -OH group showed a band in the range (3225-3390 cm<sup>-1</sup>). Compounds containing the -NO<sub>2</sub> group showed bands in the range (1357-1330 cm<sup>-1</sup>) (symmetrical stretching) and at (1509-1528 cm<sup>-1</sup>) (asymmetric stretching). The IR and CHN data of the prepared compounds are shown in (Table 3). The <sup>1</sup>H-NMR of the prepared chalcones showed generally bands in the range 7.6-8.2 ppm due to the olefinic protons which are conjugated with the carbonyl group while the phenolic compounds showed the -OH bonds in the range 9.6-10.2 ppm [13]. The <sup>13</sup>C-NMR for the prepared compounds generally showed a band between 187.6-189.5 ppm due to the carbon of carbonyl group and carbon (B) in the range 126.8-118.2 ppm and carbon (C) in the range 139-146.6 [14]. The <sup>1</sup>H and <sup>13</sup>C-NMR data for compounds 4-14 are shown in (Table 4).



**Scheme 1:** Synthesis of Chalcones 4-14



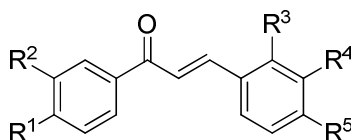
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**Scheme 2:** Dowex resin, Dowex resin (1), (2), and (3).

**Table 1:** Physical properties of the chalcones (**4-14**) using modified Dowex (**1, 2 and 3**)



No.	Substituents	Yield % <sup>a</sup>	Colour	M.P. °C (Ref. M.P.)
4	R <sup>1</sup> =NO <sub>2</sub> , R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =H, R <sup>5</sup> =OH	37, 47, 73	Reddish-brown	185-187(204-205 <sup>15</sup> )
5	R <sup>1</sup> =NO <sub>2</sub> , R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =OH, R <sup>5</sup> =H	34, 46, 67	Pale green	195-196( <sup>b16</sup> )
6	R <sup>1</sup> =H, R <sup>2</sup> =NO <sub>2</sub> , R <sup>3</sup> =H, R <sup>4</sup> =H, R <sup>5</sup> =OH	34, 49, 68	Light brown	197-200( <sup>b17</sup> )
7	R <sup>1</sup> =H, R <sup>2</sup> =NO <sub>2</sub> , R <sup>3</sup> =H, R <sup>4</sup> =OH, R <sup>5</sup> =H	32, 49, 70	Dark green	188-191( <sup>b18</sup> )
8	R <sup>1</sup> =NO <sub>2</sub> , R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =H, R <sup>5</sup> =H	33, 47, 71	Yellow	143-144(146 <sup>11</sup> )
9	R <sup>1</sup> =H, R <sup>2</sup> =NO <sub>2</sub> , R <sup>3</sup> =H, R <sup>4</sup> =H, R <sup>5</sup> =H	38, 51, 76	Yellow	129-131(130-131 <sup>18</sup> )
10	R <sup>1</sup> =H, R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =OH, R <sup>5</sup> =H	37, 48, 74	Dark brown	191-193(199-200 <sup>19</sup> )
11	R <sup>1</sup> =H, R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =H, R <sup>5</sup> =OH	34, 46, 71	Bronze	192-194(185-187 <sup>20</sup> )
12	R <sup>1</sup> =H, R <sup>2</sup> =H, R <sup>3</sup> =NO <sub>2</sub> , R <sup>4</sup> =H, R <sup>5</sup> =H	34, 46, 76	Yellow	126-130(125 <sup>21</sup> )
13	R <sup>1</sup> =H, R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =NO <sub>2</sub> , R <sup>5</sup> =H	36, 50, 79	Yellow	139-141(124 <sup>12</sup> )
14	R <sup>1</sup> =H, R <sup>2</sup> =H, R <sup>3</sup> =H, R <sup>4</sup> =H, R <sup>5</sup> =NO <sub>2</sub>	35, 49, 77	Yellow	157-159(158-160 <sup>22</sup> )

a) % Yields based on Dowex Resins (**1, 2 and 3**) respectively.

b) No MP was reported for this compound in the reference mentioned.

**Table 2:** Determination of %SO<sub>3</sub>H in the resins by titration with base

DS%	SO <sub>3</sub> H%	SO <sub>3</sub> H (g)	No of Milliequiv. Consumed	Dowex Resin and Modified Dowex
16.165	4.43	0.04438	0.55	Acidified Dowex by HCl (50%)
-----	5.72	0.05729	0.71	Nitrated Dowex
53.984	11.78	0.11782	1.46	Sulfonated Dowex

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**Table 3:** IR and C.H.N analyses of compounds (**4-14**)

No	IR (KBr)/Cm <sup>-1</sup>	CHN analyses					
		%C Calculated	%C Found	%H Calculated	%H Found	%N Calculated	%N Found
4	1336 (sym. NO <sub>2</sub> ), 1514(assy. NO <sub>2</sub> ), 1558 (Ar), 1610 (C=C), 1649(C=O), 2232 (OH)	66.91	66.1	4.12	5.17	5.20	5.17
5	1344 (sym.NO <sub>2</sub> ), 1450 (Ar), 1515 (assy.NO <sub>2</sub> ), 1575(C=C), 3265 (OH)	66.91	66.1	4.12	4.88	5.20	5.30
6	1347 (sym.NO <sub>2</sub> ), 1515 (assy.NO <sub>2</sub> ), 1562( Ar), 1606(C=C), 1657(C=O), 3236 (OH)	66.91	66.1	4.12	5.10	5.20	5.31
7	1350 (sym.NO <sub>2</sub> ), 1453 (Ar), 1521 (assy.NO <sub>2</sub> ), 1588 (C=C), 1656 (C=O), 3390 (OH)	66.91	66.1	4.12	4.48	5.20	5.28
8	1330 (sym.NO <sub>2</sub> ), 1453 (Ar), 1514 (assy.NO <sub>2</sub> ), 1596 (C=C), 1658 (C=O)	71.14	71.20	4.38	4.50	5.53	5.41
9	1339 (sym.NO <sub>2</sub> ), 1481 (Ar), 1521 (assy.NO <sub>2</sub> ), 1598 (C=C), 1660 (C=O)	71.40	71.02	4.38	4.76	5.53	5.43
10	1577 (Ar), 1602 (C=C), 1650 (C=O), 3328 (OH)	80.34	80.42	5.39	5.51	-	-
11	1505 (Ar), 1553 (C=C), 1652 (C=O), 3225 (OH)	80.34	80.55	5.39	5.55	-	-
12	1338 (sym.NO <sub>2</sub> ), 1512 (assym.NO <sub>2</sub> ), 1574 (Ar), 1603 (C=C), 1666 (C=O)	71.14	71.31	4.38	4.52	5.53	5.33
13	1344 (sym.NO <sub>2</sub> ), 1523 (assy.NO <sub>2</sub> ), 1571 (Ar), 1602 (C=C), 1664 (C=O)	71.14	71.40	4.38	4.49	5.53	5.48
14	1338 (sym.NO <sub>2</sub> ), 1453 (Ar), 1514 (assy.NO <sub>2</sub> ), 1605 (C=C), 1656 (C=O)	71.14	71.27	4.38	4.42	5.53	5.43

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**Table (4):**  $^1\text{H}$  and  $^{13}\text{C}$ -NMR data of the chalcones (**4-14**)

NO	$^1\text{H}$ -NMR (DMSO- $d_6$ , ppm)	$^{13}\text{C}$ -NMR (DMSO- $d_6$ , ppm)
4	6.85(2H, s, Ar-H), 7.74-7.79(2H, d, $J = 15.9\text{Hz}$ , CH=CH), 8.3-8.33(4H, m, Ar-H), 10.21(1H, s, OH).	188.0, 161.2, 150.3, 146.1, 143.4, 131.4, 131.1, 130.4, 126.4, 126.2, 124.4, 124.1, 118.2, 116.3, 116.1
5	6.89(2H, s, Ar-H), 7.27-7.32(2H, d-d, $J = 15.9\text{Hz}$ CH=CH), 8.36-8.39(4H, m, Ar-H), 9.69(1H, s, OH),	188.2, 158.4, 150.3, 146.2, 142.4, 136.2, 130.4, 130.1, 130.0, 124.4, 124.1, 122.0, 120.2, 118.3, 115.4
6	6.87(2H, m, Ar-H), 7.8-7.89(CH=CH, Ar-H), 8.49-8.80(4H, m, Ar-H)	187.4, 161.0, 150.2, 146.1, 142.3, 131.1, 130.4, 130.2, 130.0, 127.4, 124.3, 124.0, 120.4, 118.2, 116.4
7	6.91(2H, m, Ar-H), 7.3(2H, m, Ar-H), 7.7-7.83(3H, m, CH=CH and Ar-H), 8.45-8.56(2H, m, Ar-H), 8.7(1H, s, OH), 9.64(1H, s, OH)	187.3, 158.4, 148.4, 140.3, 139.4, 136.2, 135.0, 130.3, 130.1, 127.0, 123.4, 121.4, 120.2, 118.3, 115.4
8	7.48(5H, s, Ar-H), 7.82-7.98(2H, m, CH=CH), 8.36-8.41(4H, m, Ar-H)	188.1, 150.4, 146.2, 142.4, 124.2, 142.0, 134.3, 131.4, 131.1, 130.3, 130.0, 129.4, 129.1, 128.9, 122.2
9	7.48(5H, s, Ar-H), 7.84-8.01(2H, m, CH=CH), 7.92-8.61(3H, m, Ar-H), 8.84(1H, s, Ar-H)	189.5, 160.0, 144.8, 138.4, 133.2, 131.4, 131.1, 129.3, 129.0, 128.1, 126.2, 126.1, 118.3, 116.4, 116.1
10	6.87(1H, m), 7.22(1H, br, s), 7.33-7.24(2H, m), 7.67(1H, d, $J = 15.6\text{Hz}$ ), 7.69-7.53(3H, m), 7.87(1H, d, $J = 15.6\text{Hz}$ ), 8.15-8.11(2H, m), 9.67(br, 1H, OH)	189.2, 160.2, 145.3, 138.4, 133.0, 131.4, 131.1, 129.1, 129.1, 128.3, 126.1, 126.3, 118.2, 116.4, 116.4
11	6.85(2H, m, Ar-H), 7.54(2H, m, Ar-H), 7.73(5H, s, Ar-H), 7.76(2H, m, Ar-H), 10.16(1H, s, OH)	189.3, 160.2, 145.4, 138.4, 133.2, 131.4, 131.1, 129.1, 129.0, 128.3, 126.2, 126.1, 118.3, 116.4, 116.1
12	7.70-7.89(5H, m, Ar-H), 7.92(H, d, $J = 15.9\text{Hz}$ , CH=CH), 7.99(2H, m, Ar-H), 8.1(1H, m, Ar-H), 8.21(1H, d, $J = 15.9\text{Hz}$ , CH=CH)	189.3, 149.4, 139.4, 137.3, 134.0, 133.4, 131.1, 130.4, 130.4, 129.4, 129.3, 129.1, 129.0, 126.1, 125.4
13	7.58(3H, m, Ar-H), 7.69-7.82(2H, m, Ar-H), 7.87(2H, m, Ar-H), 8.14-8.19(2H, d, $J = 15.9\text{Hz}$ , CH=CH), 8.32(1H, m, Ar-H), 8.77(1H, s, Ar-H)	189.3, 148.4, 141.0, 137.4, 137.2, 135.2, 133.3, 130.1, 130.0, 129.3, 129.1, 129.0, 125.3, 125.1, 123.4
14	7.71-7.48(4H, m), 7.79(2H, d, $J = 2\text{H}$ , d, $J = 8.8\text{Hz}$ ), 7.83(1H, d, $J = 15.8\text{Hz}$ ), 8.04(2H, m), 8.28(2H, d, $J = 8.8\text{Hz}$ )	189.6, 141.5, 141.0, 137.5, 133.4, 133.3, 128.9, 128.8, 128.7, 128.6, 128.5, 125.7, 125.5, 124.2, 124.0

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### 3. Experimental

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#### 3.1. Materials and Instruments:

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Melting points were recorded on electrothermal gallenkamp apparatus and uncorrected. IR spectra were recorded using KBr on Shimadzu 8400 S-FT-IR spectrophotometer.  $^1\text{H}$  and  $^{13}\text{C}$ -NMR were recorded at Al-Bayt University, Jordan using a Bruker, Vitra Shield 300MHz instrument with  $\text{DMSO-d}_6$  as solvent and TMS as internal standard. Elemental analyses were also carried out at the above university.

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#### 3.2. Modification of Dowex [10]:

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Cross linked sodium salt form Dowex (8%) with 20-50 mesh was used in this study and it was modified as follows:

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a) **Conversion into acid form (1):** Hydrochloric acid (50%, 200 mL) was added to (20 g) of the resin (which was pre-dried at  $70^\circ$  for 24h) in a 250 mL round bottomed flask and the mixture was heated for 2h with stirring in a water bath at  $50^\circ\text{C}$ . The resin was then filtered, washed extensively with distilled water until all the acidic material was removed and then washed with small amount of methanol. After drying the weight of the recovered material was 16.9g.

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b) **Nitration of Dowex (2):** Concentrated nitric acid (47 mL) and concentrated sulfuric acid (51mL) were mixed cautiously with cooling in an ice bath, and then 20 g of Dowex was added stepwise. The mixture was heated in a water bath at  $70^\circ\text{C}$  for 7h with stirring, and then the mixture was added carefully with stirring over a period of 30 min to distilled water (250 mL). The resin was filtered, washed with distilled water and dried as above. The weight of the recovered material was 24.2 g.

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c) **Sulfonation of Dowex (3):** Concentrated sulfuric acid (100 mL) was added to 20 g of dried resin then the mixture was heated for 18 h with stirring in a water bath at  $90^\circ\text{C}$ . The mixture was added to distilled water (400 mL) in a beaker and stirred for 15 min; the resin was then filtered, washed and dried as above. The weight of the recovered material was 24.1 g. The structure of the (acidic forms) of the prepared resins are shown in (Table 1).

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#### 3.3. Preparation of Chalcones (4-14):

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The modified resins (0.50 g) (1, 2, and 3) were added to the ketone dissolved in absolute ethanol (5 mL) and the mixture was stirred at room temperature for 5 min. The reaction mixture was added to the aldehyde solution in (5 mL) of absolute ethanol, and then the resultant mixture was heated under reflux for 4 h. The hot reaction mixture was filtered immediately to separate the insoluble resin, then the filtrate was cooled and the precipitate was filtered, recrystallized from absolute ethanol (10 mL) and washed with cold ether. All the reacted aldehydes and ketones were reacted in equimolar rations as shown in (Table 2).

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#### 3.4. Determination of % $-\text{SO}_3\text{H}$ in the Resins:

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The dried resin (1.00 g) containing  $-\text{SO}_3\text{H}$  group was treated with distilled water (20 mL) for swelling,

then it was treated with hydrochloric acid (0.1N, 20 mL) for 24 h. Following this it was transferred into a separating funnel and the acid aqueous layer was removed. The resin was washed extensively with distilled water until pH 7. Sodium hydroxide solution (0.1N, 15 mL) was added to the acidified resin, then the excess basic solution was removed and collected in conical flask and the resin in the funnel was further washed with distilled water until all the base was removed. The whole basic solution was then titrated with hydrochloric acid (0.1N) in the presence of methyl orange as indicator. The % -SO<sub>3</sub>H weight of the resin could be determined from the following equation:

$$wt\ of\ -SO_3H = \frac{M.\ wt\ of\ -SO_3H \times (N)_{NaOH} \times V_{NaOH}}{1000}$$

The results of % -SO<sub>3</sub>H of the modified resins are shown in (Table 5).

#### 4. Conclusion

The acidic modification of the Dowex resin proved to be a useful method for the synthesis of chalcones using Claisen-Schmidt condensation methodology, and in particular those bearing the nitro and hydroxyl substituents. Thus, eleven substituted chalcones were prepared using this acid medium with ease. Isolation and purification were achieved without the need for column chromatography or complex work up.

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