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A REVIEW ON SYNTHESIS OF CHALCONE DERIVATIVES USING NON-CONVENTIONAL METHODS.

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ABSTRACT
Non-conventional methods for synthesis of different compounds have been of great interest in the organic chemistry field. In this way, some non-conventional reactions are used to prepare chalcone derivatives without some catalyst. For example, the use of energy sources such as microwave irradiation, sonochemistry and mechanochemistry. The aim of this research was to characterize some non-conventional method for synthesis of chalcone derivatives using some scientific information systems.

KEYWORDS. Chalcone, derivatives, synthesis, Non-conventional, sonochemistry, mechanochemistry

INTRODUCTION
For several years, chalcone derivatives have been used in organic chemistry and pharmaceutical industry fields[i-iv]. It is noteworthy that chalcone analogs are α-β-unsaturated ketones[v, vi] which produce different biological activity against Parkinson[vii] cancer[viii], gastric ulcer[ix], malaria[x], virus[xxi-xiv], bacteria[xv] fungi[xvi, xvii] and heart failure[xviii]. In the search of new chalcone derivatives several conventional methods have been used; for example, a study showed (Fig.1) the acetylation of 2,5-Dichloro-thiophene (1) to form 1-(2,5-dichloro-thiophen-3-yl)-ethanone (2). Then, 2 was coupling to carbaldehyde derivative in basic medium to form (*E*)-3-(5-bromothiophen-2-yl)-1-(2,5-dichlorothio- phen-3-yl)-2-propen-1-one (3)[xix].

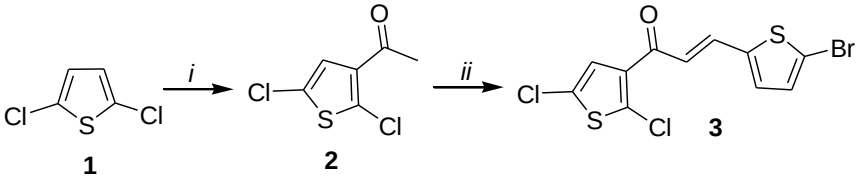


Fig. 1. Preparation of (*E*)-3-(5-bromothiophen-2-yl)-1-(2,5-dichlorothiophen-3-yl)-2-propen-1-one (**3**). *Reagents and conditions*; *i* = acetyl chloride, aluminum trichloride; *ii* = 5-Bromo-thiophene-2-carbaldehyde, NaOH.

Besides, a report (Fig. 2) display the synthesis of 1-(2,4-Dihydroxy-phenyl)-3-(2,3,4-trimethyl-phenyl)-propenone (**6**) *via* reaction of 1-(2,4-Dihydroxy-phenyl)-ethanone (**4**) with 1,2,3-Trimethyl-4-vinyl-benzene (**5**) using Montmorillonite-K-10 Clay as catalyst[xx].

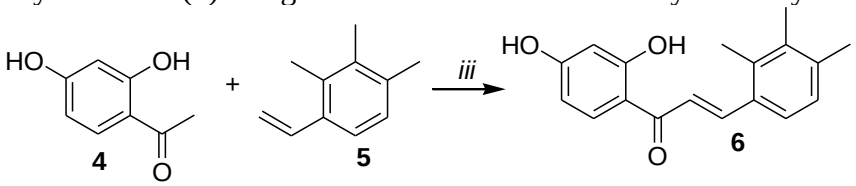


Fig. 2. Synthesis of 1-(2,4-Dihydroxy-phenyl)-3-(2,3,4-trimethyl-phenyl)-propenone (**6**). *Reagents and conditions*; *iii* = Montmorillonite K10 Clay, dimethylformamide.

Another study[xxi] shows the reaction of 1-Methyl-4-vinyl-benzene (**7**) with pinacolborane in the presence of $RhCl(cod)_2$ complex (Fig. 3) to form 2-[2-(4-Methoxy-phenyl)-vinyl]-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (**8**). Then, **8** reacted with $NaIO_4$ using the THF/H_2O system to give *p*-methoxyphenyl ethenylboronic acid (**9**). Following **9** was bound to 3,4-dimethoxy-benzoyl chloride (10) to form 3,4,4-trimethoxychalcone (**11**).

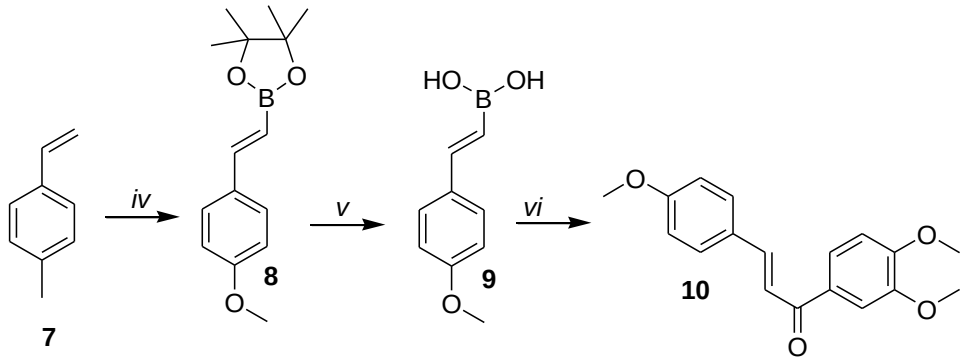


Figure 3. Synthesis of 3´,4´,4-trimethoxychalcone (**10**) via Suzuki coupling reaction. *Reagents and conditions*; *iv* = pinacolborane; *v* = $NaIO_4$, THF/H_2O; *vi* = 3,4-Dimethoxy-benzoyl chloride.

In addition, The Figure 3 display the reaction of 3-acetyl-2,5-dimethylthiophene (**11**) with Benzene-1,4-dicarbal-dehyde (**12**) in ethanolic NaOH solution to give 78% of (2*E*,2'*E*)-3,3-(1,4-Phenylene)bis[1-(2,5-dimethyl-3-thienyl)prop-2-en-1-one] (**13**)[xxii].

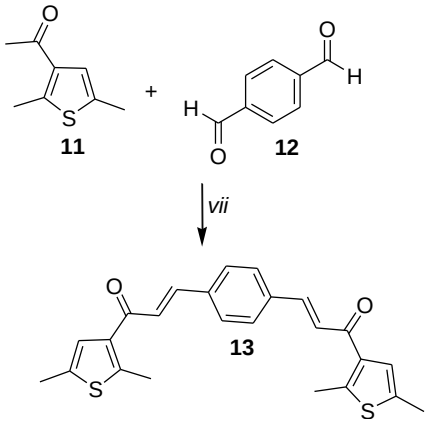


Fig. 4. Synthesis of a chalcones analog (**13**). *Reagents and conditions*; *viii* = NaOH/EtOH.

Non-conventional methods

In the literature, there are several unconventional methods for the synthesis of chalcone derivatives which have several advantages, such as *i*) reactions free catalyst; *ii*) high reaction rate and *iii*) mild reaction condition compared to conventional methods. For example, a chalcone derivative 3-(4-fluorophenyl)-1-(4-methoxyphenyl)prop-en-1-one (**16**) was prepared *via* condensation of 4-methoxyacetophenone (**14**) with 4-fluorobenzaldehyde (**15**) in ethanol (Figure 5) using solar radiation[xxiii].

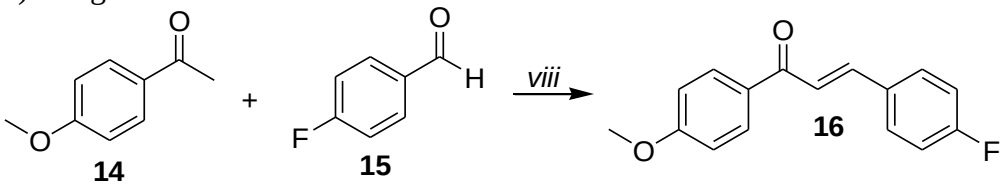


Fig. 5. Sinthesis of 3-(4-fluorophenyl)-1-(4-methoxyphe nyl) prop-en-1-one (**16**). *Reagents and conditions*; *ix* = 1-(4-Methoxy-phenyl)-ethanone, 4-Fluoro-benzaldehyde, EtOH, solar radiation.

Besides, a report showed a method[xxiv] to prepare a chalcone derivative (**19**) from 1-(5-Methyl-furan-2-yl)-ethanone (**17**), benzaldehyde (**18**) and EtOH. The reaction mixture was microwave irradiated for about 26 min at 180 watts to form **19.**

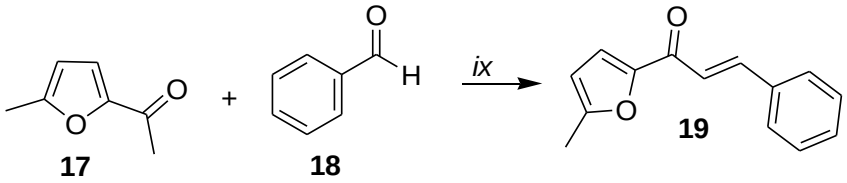


Fig. 6. Preparation of 1-(5-Methyl-furan-2-yl)-3-phenyl-propenone (**19**). *Reagents and conditions; xii* = EtOH, microwave irradiated at 180 watts.

In addition, an aromatic aldehyde (**18**), acetophenone (**20**) and potassium;oxo(oxoalumanyloxy)alumane;fluoride ($KF\text{-}Al_2O_3$) was added to an alcoholic KOH solution (Fig. 7), Then, the mixture was irradiated at 25-26 °C/25 min to obtain a chalcone derivative (**21**)[xxv] with a yield of 80 %.

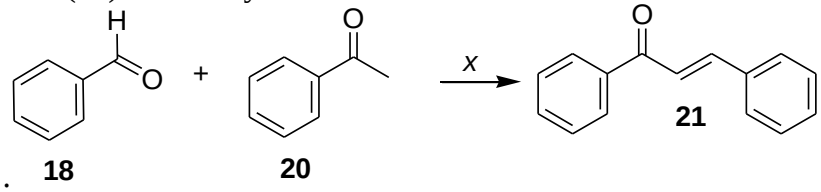


Fig. 7. Preparation of 1,3-Diphenyl-propenone (**21**). *Reagents and conditions; xii* = EtOH/KOH, KF-Al_2O_3, ultrasound irradiation.

Other study showed (Fig. 8) the synthesis of 1-(2,5-Dimethyl-thiophen-3-yl)-3-phenyl-propenone (**23**) from 3-acetyl-2,5-dimethylthio-phene (**22**) with benzaldehyde (**18**) in ethanolic NaOH using the microwave oven[xxvi].

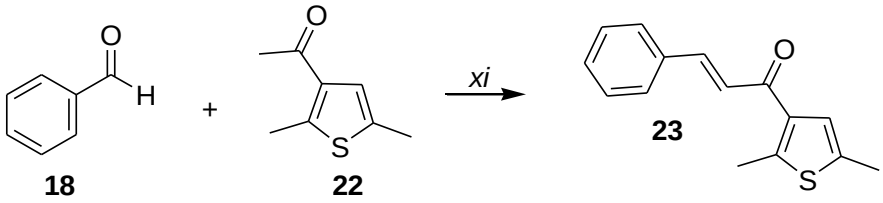
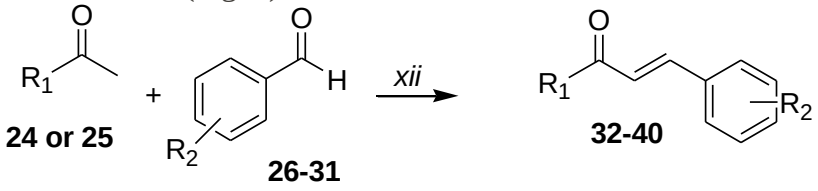


Fig. 8. Synthesis of 1,3-Diphenyl-propenone (**21**). *Reagents and conditions; xii* = EtOH/NaOH, microwave irradiation.

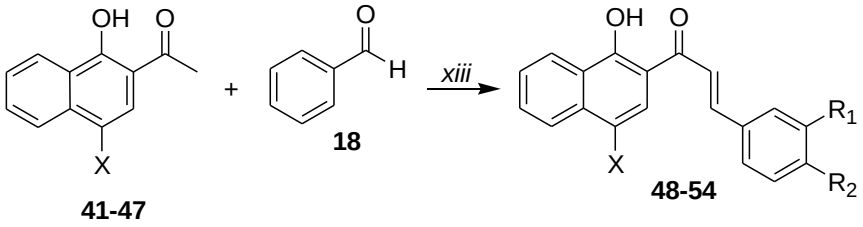
On other hand, a series of chalcone analogs (**32**-**40**) were prepared by modifying Claisen-Schmidt condensation from aryl or thiophen ketones (**24** or **25**) and aldehyde derivatives (**26**-**31**) using glycerin as solvent (Fig. 9)[xxvii].



R_1 = Phenyl or Thiophen
R_2 , 4-OCH_3; 4-F; 4-BR;4-Cl; 4-CH_3; 3-OCH_3

Fig. 9. Synthesis of chalcones derivatives (**19**-**27**). *xii* = glycerin.

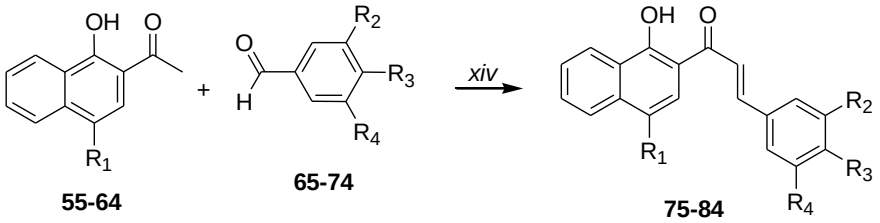
In addition, some chalcone analogs (**48**-**54**) were prepared from 2-acetyl-1-naphthol or their halo-derivatives (**41**-**47**) and benzaldehyde (**18**). It is noteworthy that reaction mixture was microwave irradiated for about 2-5 min at 180 W (Fig. 10)[xxviii].



X = H, Br, I, Cl, H, Br, I
R_1 = OCH_3
R_2 = OBn

Fig. 10. Preparation of chalcone analogs (**48**-**54**). *Reagents and conditions*; *xiii* = EtOH/KOH, microwave irradiated (180 W).

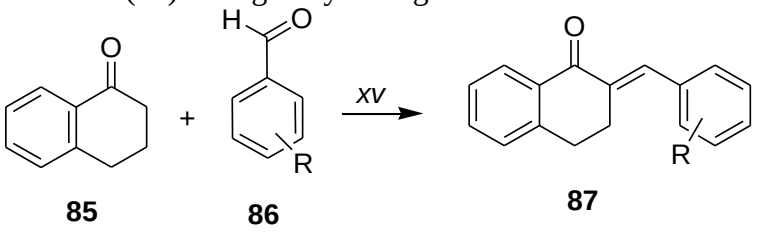
Besides, Zangade in 2011 developed a series of chalcone derivatives (**75**-**84**) *via* reaction of 2-substituted acetyl-1-naphthol/2-acetyl-1-naphthol (**55**-**64**) and some substituted benzaldehyde derivatives (**65**-**74**) in the presence of potassium hydroxide using grinding technique[xxix]; it is noteworthy that this type of reaction does not need catalysts, giving excellent performance in a short reaction time (Fig.11).



75 (R_1, R_2 = H; R_3, R_4 =OCH_3)
76 (R_1, R_2, R_4 = H; R_3 = 0CH_3)
77 (R_1 = I; R_2, R_4 = H; R_3 OCH_3)
78 (R_1 = Br; R_2 = H, R_3, R_4 = OCH_3)
79 (R_1 = Br; R_2, R_3, R_4 = OCH_3
80 (R_1, R_2, R_4 = H; R_3 = Cl)
81 (R_1 = I, R_2, R_4 = H; R_3 =Cl)
82 (R_1 = Br; R_2, R_4 = H; R_3 F)
83 (R_1, R2, R_4 = H; R_3 =F)
84(R_1 = H; R_2, R_3, R_4 = OCH_3

Fig. 11. Synthesis of a series of chalcone derivatives (**75-84**). *Reagents and conditions*; *xiv* = KOH/ grinding.

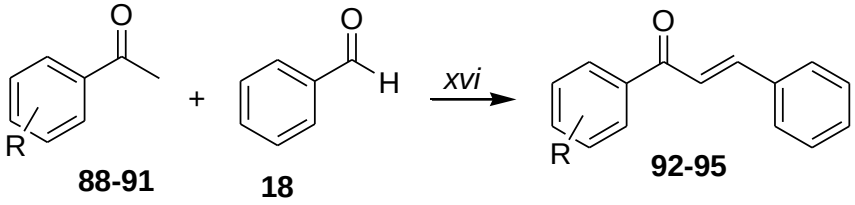
Another report (Fig. 12) showed the reaction of an aldehyde derivative (**86**) with 3,4-Dihydro-2H-naphthalen-1-one in the presence (**85**) of Eaton's reagent under microwave irradiation to form a chalcone derivative (**87**) with good yielding in short reaction times of 3-5 min[xxx].



R = -OCH_3

Fig. 12. Synthesis of a series of chalcone derivatives (**75-84**). *Reagents and conditions*; *xv* = Eaton's reagent; microwave irradiation, 3-5 min.

On the other hand, some chalcone derivatives (Fig. 13) have been prepared (**92-95**) from ketone derivatives (**88-91**) and benzaldehyde (**18**) using activated carbons (Na- and Cs-Norit) and sonochemical irradiation[xxxi].



R = H, 3-NO_2, 4-Cl, 2,4-di-Cl

Fig. 13. Synthesis of chalcone derivatives (**92-95**). *Reagents and conditions; xvii* = Na- and Cs-Norit/carbon, EtOH.

Recently, was prepared a chalcone derivative (**21**) via reaction of aryl ketone (**20**) and benzaldehyde (**18**) using amino zeolites (solid phase) under ultrasonic irradiation (Fig. 14). It is noteworthy that with this procedure the chalcone can be selectively obtained with high yield[xxxii, xxxiii].

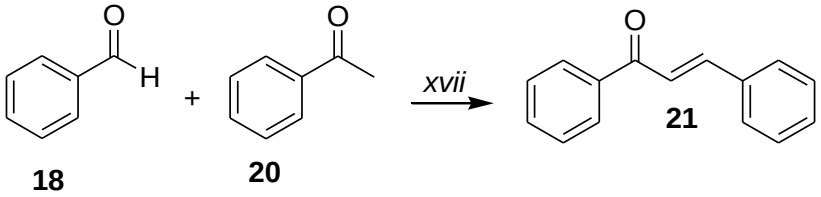
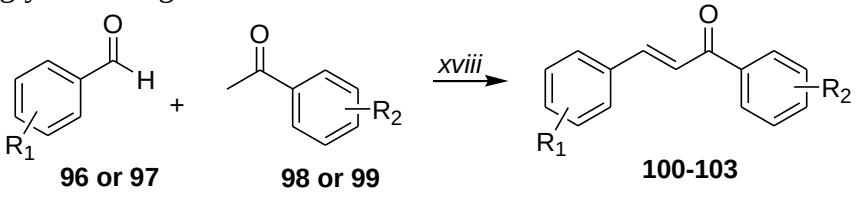


Fig. 14. Preparation of a chalcone derivative (**21**). *Reagents and conditions; xvii* = NaOH, amino-zeolites.

On the other hand, a study (Fig. 15) showed the preparation of some chalcone derivatives (**100-103**) from aldehyde derivatives (**96** or **97**) and ketone analogs (**98** or **99**) (Fig.15) using zincferrite nanoparticules ($ZnFe_2O_4$) which were developed by sol-gel auto burning by utilization of glycine as a green fuel[xxxiv].



R_1 = -Cl; -NO_2
R_2 = -Cl: -OCH_3

Fig. 15. Preparation of chalcone derivatives (**100-103**). *Reagents and conditions; xviii* = $ZnFe_2O_4$, glycine.

Furthermore, a report (Figure 16) showed the reaction of 4-Nitro-benzaldehyde (**97**) with acetophenone (**20**) to form 3-(4-Nitro-phenyl)-1-phenyl-propenone (**104**) using activated carbon with higher yielding[xxxv].

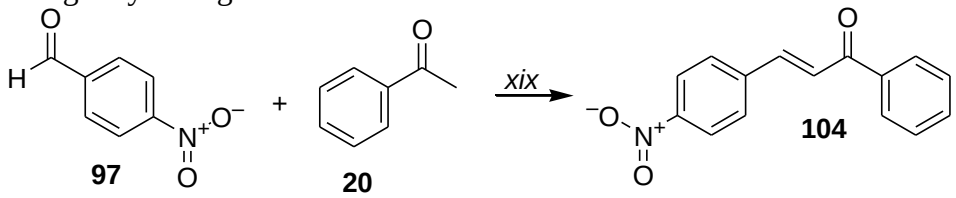
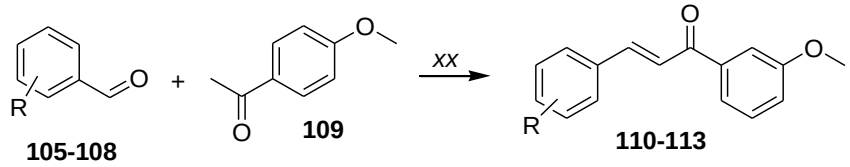


Fig. 16. Preparation of -(4-Nitro-phenyl)-1-phenyl-propenone (**67**). *Reagents and conditions; xix* = activated carbon.

Other data (Fig. 17) indicate that hydroxyapatite can be used as an inert support for the synthesis of chalcone derivatives; for example, compounds **110-113** were prepared from aldehyde analogs (**105-108**) and 1-(4-Methoxy-phenyl)-ethanone (**109**) in the presence of Hydroxyapatite and microwave irradiation[xxxvi].



R = *p*-Cl, *m*-NO_2, *p*-OCH_3, *p*-OH

Fig. 17. Preparation of chalcone derivatives (**110-113**). *Reagents and conditions; xx* = hydroxyapatite, microwave irradiation (700 W), 5 min.

Other study (Fig. 18) showed the synthesis of 1-(3,5-Dibromo-2,4-dihydroxy-phenyl)-3-phenyl-propenone (**115**) via reaction of 1-(3,5-Dibromo-2,4-dihydroxy-phenyl)-ethanone (**114**) with benzaldehyde (**18**) in the presence of basic alumina and microwave irradiation for 4-6 min[xxxvii].

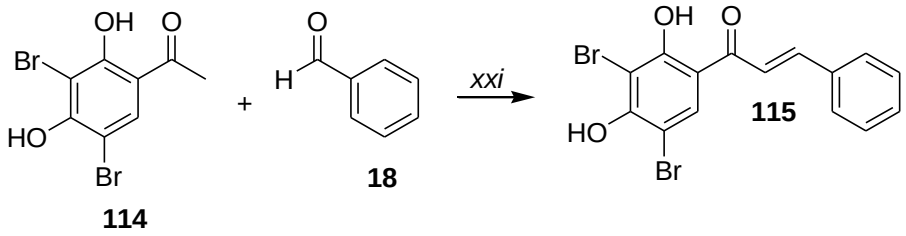


Fig. 18. Synthesis of 1-(3,5-Dibromo-2,4-dihydroxy-phenyl)-3-phenyl-propenone (**115**). *Reagents and conditions; xxi* = basic alumina/DMF, microwave irradiation (700 W).

Another report (Fig. 19) shows the synthesis of chalcone derivatives (116 and 117) from benzaldehyde (18) and acetophenone (20) in the presence of an aluminate mesoporous silica nanomaterial solvent-free and low temperature conditions. The proposed mechanism describes the formation of an enolate as intermediary *via* formation of bonds between the carbonyl groups and the oxygen atoms of the Al-O bands[xxxviii].

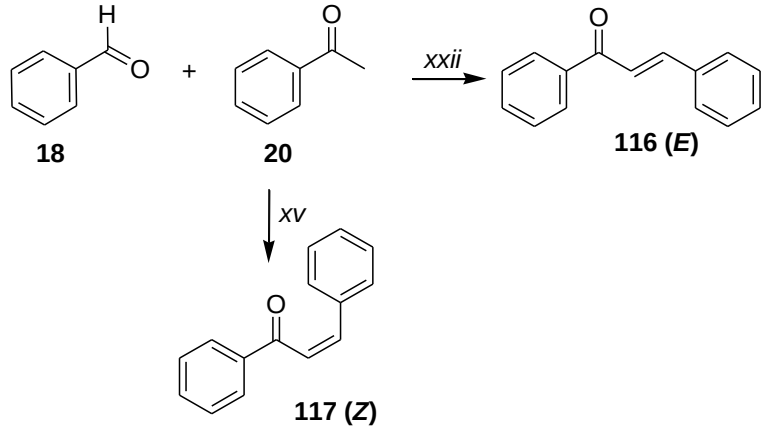


Fig. 19. Preparation of chalcone derivatives (**116**, **117**). *Reagents and conditions; xxii* = aluminate mesoporous silica/room temperature.

Other study (Fig. 20) shows the synthesis of 1,3-Diphenyl-propenone (**21**) from benzaldehyde (**18**) and acetophenone (**20**) in the presence of $–SO_3H$-functionalized ionic liquids or 1-Butyl-2,3-dimethylimidazolium bromide as a non-conventional chemical alternative for the synthesis of chalcone derivatives[xxxix].

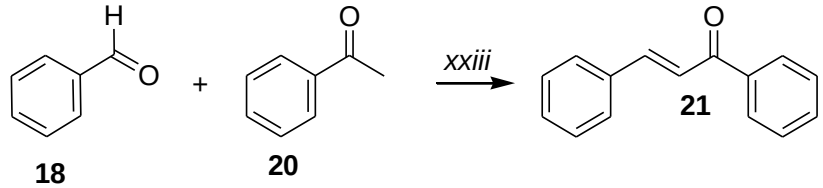


Fig. 20. Preparation of 1,3-Diphenyl-propenone (**21**). *Reagents and conditions; xxiii* = $–SO_3H$-functionalized ionic liquids or 1-Butyl-2,3-dimethylimidazolium bromide

Besides, the chalcone (**21**) was prepared via reaction of acetophenone (**20**) and benzaldehyde (**18**) using bamboo char sulfonic acid under solvent-free condition (Fig. 21). It is noteworthy that solid acid catalyst was prepared from bamboo sawdust via sulfuric acid charring[xL].

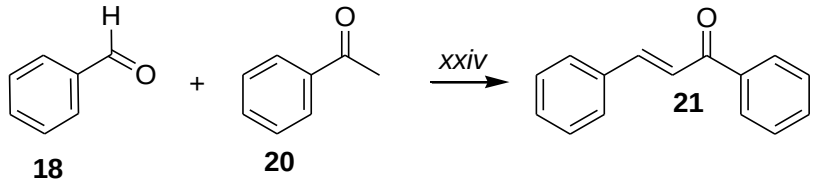


Fig. 21. Preparation of 1,3-Diphenyl-propenone (**21**). *Reagents and conditions; xxiv* = bamboo char sulfonic acid.

Finally, another study (Fig. 22) showed the condensation of acetophenone (**20**) with benzaldehyde (**18**) in the presence of Al-Mg hydrotalcites to give the compound **21** (trans-chalcone). The mechanism generally involves the formation of the anion of acetophenone followed by its attack to the carbonyl group of benzaldehyde[xLi].

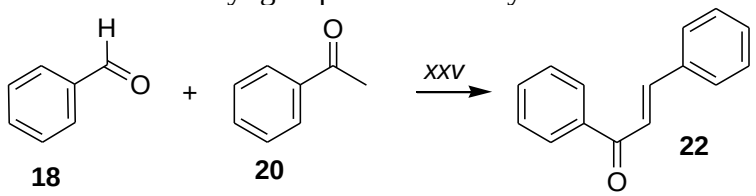


Figure 22. Synthesis of *trans*-chalcone (**22**). *Reagents and conditions; xxv* = Al-Mg hydrotalcite,

CONCLUSIONS

This review describes the synthesis of various chalcone derivatives using some unconventional methods that are of great interest in the field of organic chemistry. These protocols do not involve the use of specific catalysts. In addition, the reactions are carried out through the use of some energy sources such as microwave radiation, sonochemistry and mechanochemistry.

ACKNOWLEDGEMENT

None

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