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AN OVERVIEW OF QUANTITATIVE AND QUALITATIVE APPROACHES ON THE SYNTHESIS OF HETEROCYCLIC KOJIC ACID SCAFFOLDS THROUGH THE MULTI-COMPONENT REACTIONS

Fatemeh Mohajer and Ghodsi Mohammadi Ziarani*

Department of Chemistry, Alzahra University, Vanak Square, 1993893973, Tehran, Iran, gmohammadi@alzahra.ac.ir

Abstract – Multi-component reactions as powerful synthetic methods were developed to provide efficient complex scaffolds, including kojic acid, through the one-pot one-step fashion. This review highlights the progress of multicomponent reactions covering kojic acid under different conditions through, short reaction time, higher yields, and environmental friendliness *via* producing various molecules. The aim of this paper is to review the literature from 2015 to 2020 *via* quantitative and qualitative approaches.

CONTENTS

1. INTRODUCTION

In 1912, Yabuta described the steamed rice were inoculated with *Aspergillus oryzae* and some acid was extracted through petroleum ether named koji acid. In 1916, Yabuta used the name kojic acid, and

corrected the molecular formula to $C_6H_6O_4$.[1] Saito[2] discovered kojic acid, as the demanding inhibitor of tyrosinase, which was used in food such as crab, shrimp, and vegetables in the food industry due to antioxidant activity and natural antibiotic.[3,4] Kojic acid is a fungal metabolite which was produced by various species such as *Aspergillus*, *Acetobacter*, and *Penicillium*. Another source of kojic acid is through fermentation of glucose, sucrose, acetate, ethanol, arabinose, and xylose because of carbon sources by *Aspergillus falavus*.[5,6] Another source of kojic acid or 5-hydroxy-2-hydroxymethyl-(4*H*)-pyran-4-one is from leaves of common bearberry, to protect skin *via* lightening properties.[7] Among all compounds of kojic acid scaffold, pyranopyranes as a fused oxygenated structures[8] are one of the essential classes which have biological activities, like antibacterial,[9] anti-cancer,[10] antianaphylactic.[11] The kojic acid derivativrs have many applications in cosmetic,[12] medicine,[13] food,[14] agriculture,[15] and chemical productions.[16] Kojic acid **1** is a well-known tyrosinase inhibitor,[17] due to its structural similarity to phenolic substrates.[18] This compound could chelate copper through the active site of the enzyme; therefore, the reasonable inhibitory effect has been expected.[19] There are most commercially available tyrosinase inhibitors such as arbutin **2** and hydroquinone **3**, which were shown in Figure 1. In terms of natural resources limititation, it is necessary to be synthesized.[4]

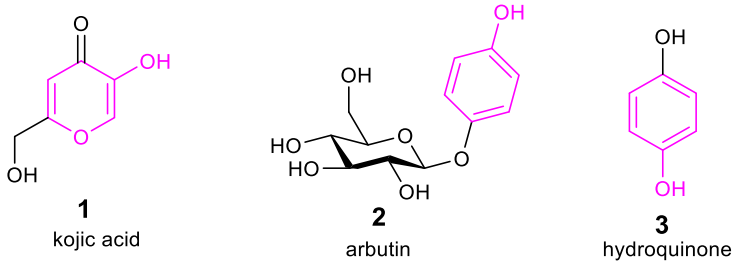

Figure 1. The structure of some tyrosinase inhibitors

The three-component reaction of kojic acid, aldehyde or 1,3-dicarbonyl motifs, and malononitrile is one of the most important process to provide heterocyclic motifs using different catalysts such as $InCl_3$,[20] CAN,[21] Al_2O_3,[22] $Bi(OTf)_3$,[23] $CeCl_3{\cdot}7H_2O/SiO_2$,[24] $FeCl_3$-SiO_2,[25] $Fe_3O_4@SiO_2$,[26] imidazole,[27] piperidine,[28] Et_3N,[29] and NH_4VO_3.[30] This reaction can be accomplished under ultrasonic irradiation.[32] In continuous our previous work[32-35] in multicomponent reaction in organic compounds, kojic acid was reviewed through quantitave and qualitative approaches.

Based on Scopus database, there are about 288 research papers related to kojic acid from 2015-2020, which demonstrates the publication rate of kojic acid. The chart of documents related to kojic acid by subject area was shown in Figure 2.

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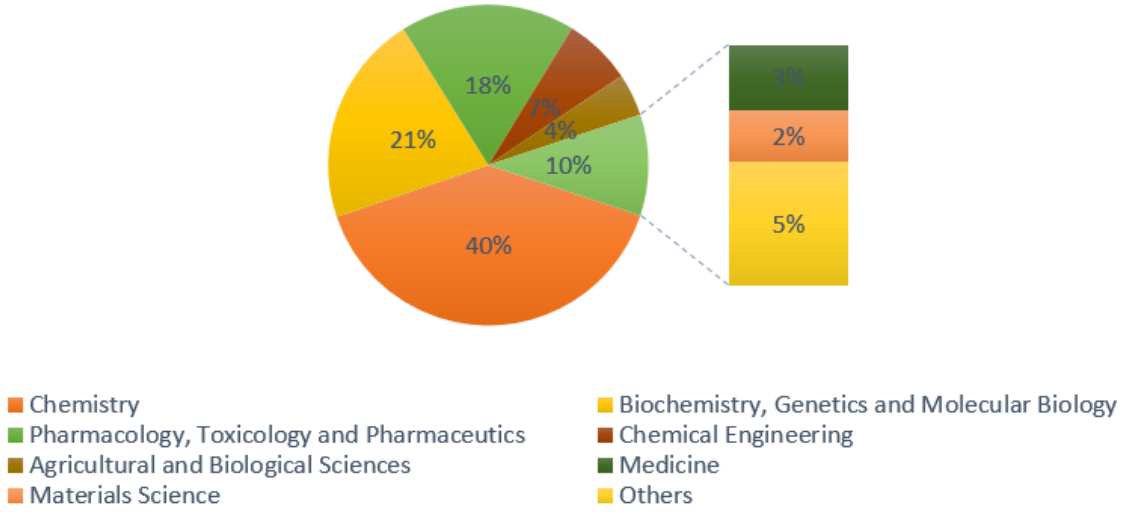


Figure 2. The chart of documents related to kojic acid by subject area

The data based on source title about kojic acid shows that 21 published papers in Bioorganic Chemistry and take the first rank among others which shows the biological activities of kojic acid derivatives (Figure 3).

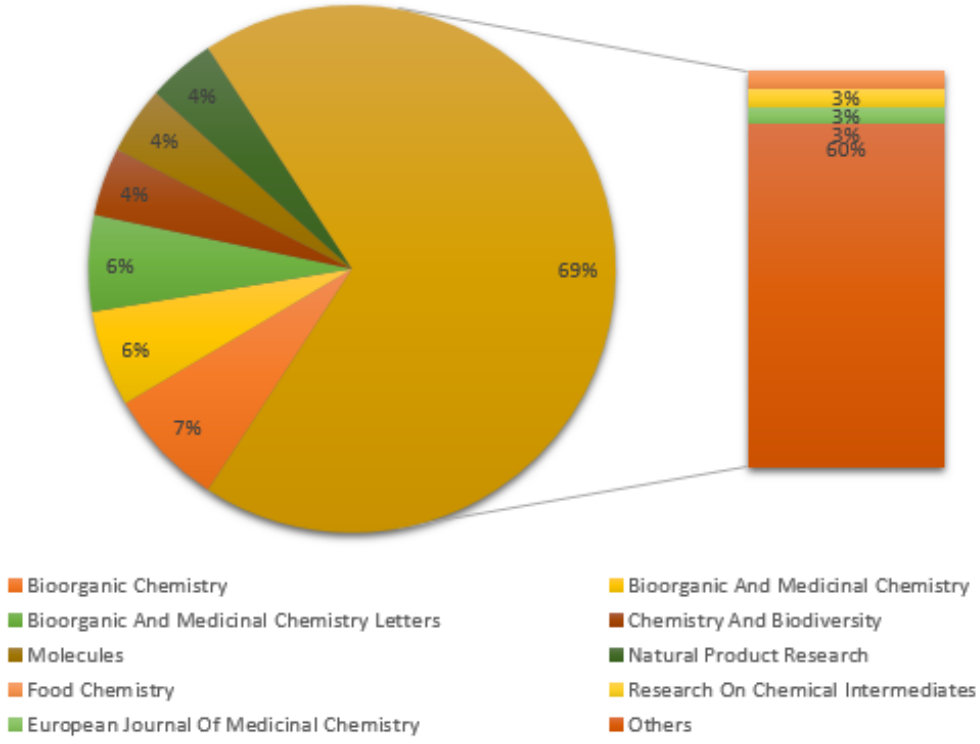


Figure 3. The chart of documents related to kojic acid by source title

Three-Fields Plot on the “kojic acid” research area was shown in Figure 4, which demonstrated the relationship title (right column), top keywords plus (middle column), and abstract (left column). The words accounted as Keywords Plus are words or phrases that frequently appear in the titles of an article’s references, and appear in the title of the article itself. The keywords plus in Figure illustrates “kojic acid,” “tyrosinase inhibitor”, “synthesis” or “molecular docking” which papers in middle column. The attractive keyword plus for the title of the selected papers are “kojic acid”, “synthesis”, “derivatives”, “tyrosinase”, “activity”, “inhibitors”, “molecular”. In the abstract column, kojic acid as a keyword was selected in all papers with the title of kojic, tyrosinase, inhibitors, synthesis with tyrosinase and kojic, and acid due to the strong relation between tyrosinase and kojic acid as an inhibitor.

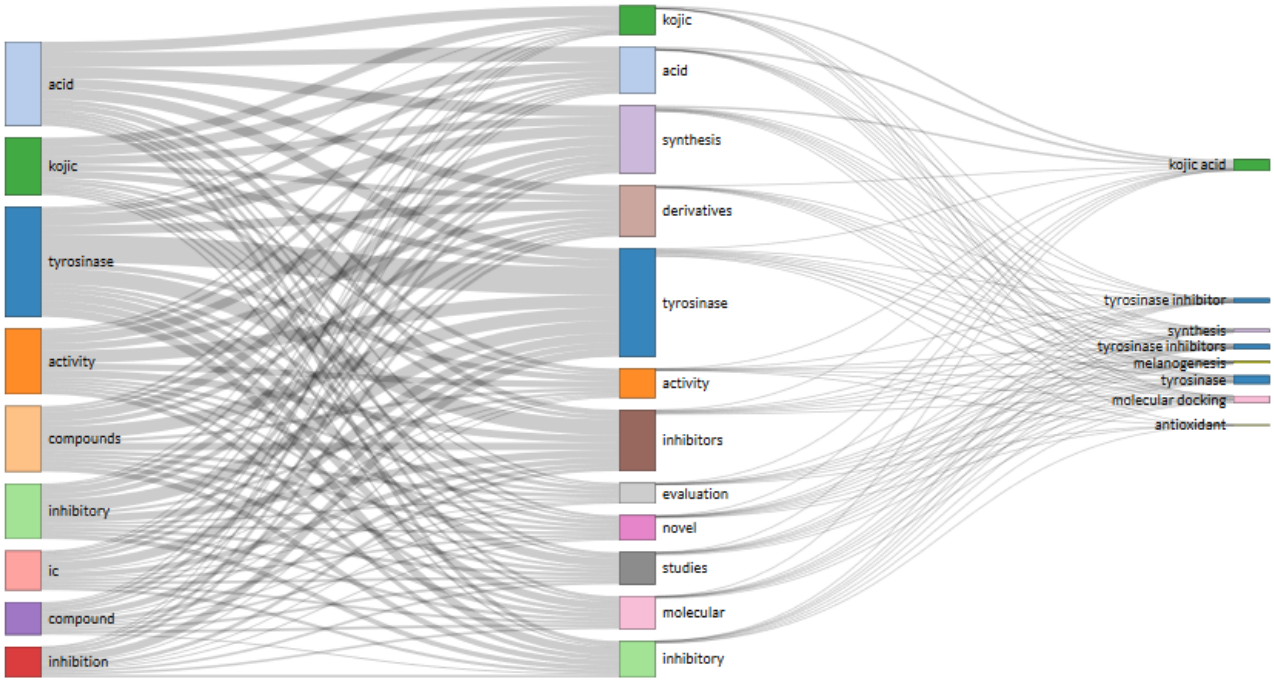


Figure 4. Three-Fields Plot of Top abstracts, Top Keywords Plus, and Top titles on “Kojic Acid.”

The evolution of the title words in “kojic acid” from 2015 to 2020 as shown in Figure 5 indicates a clear increasing trend of the title words in the bioorganic chemistry by the time.

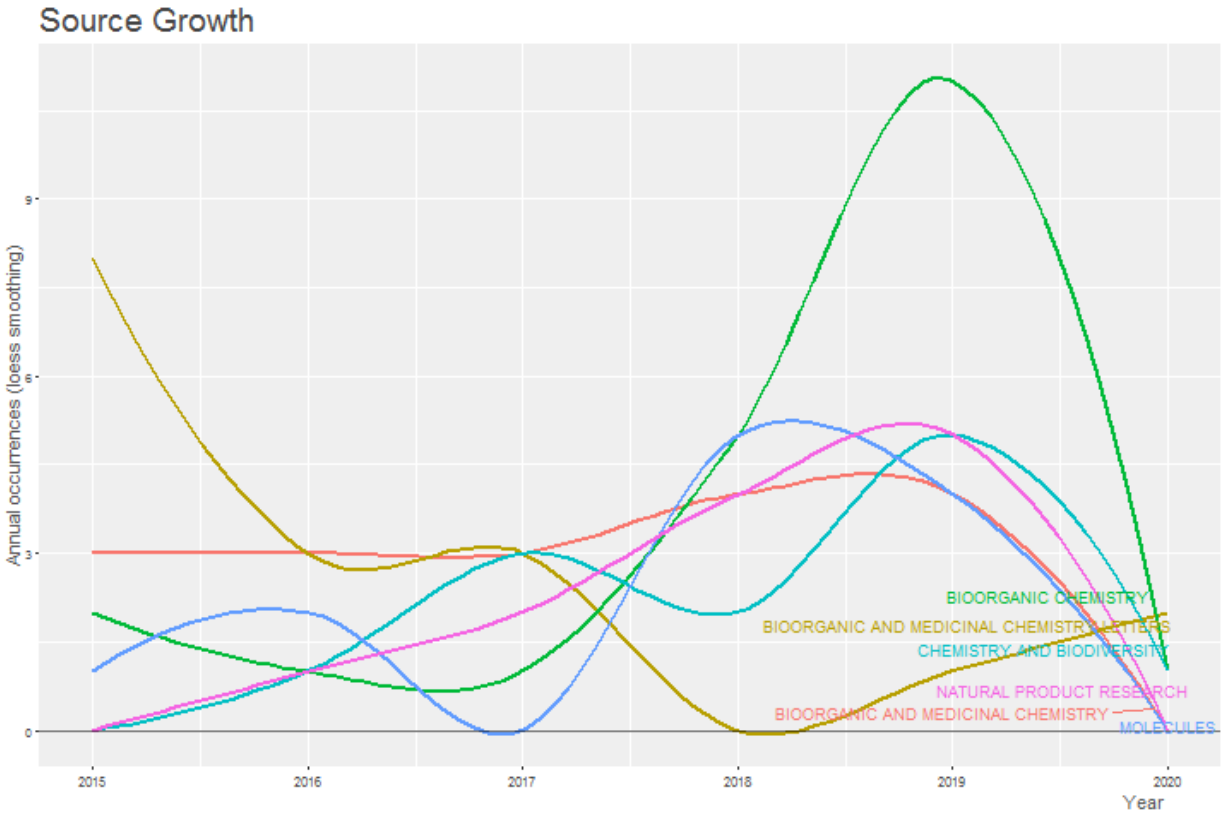
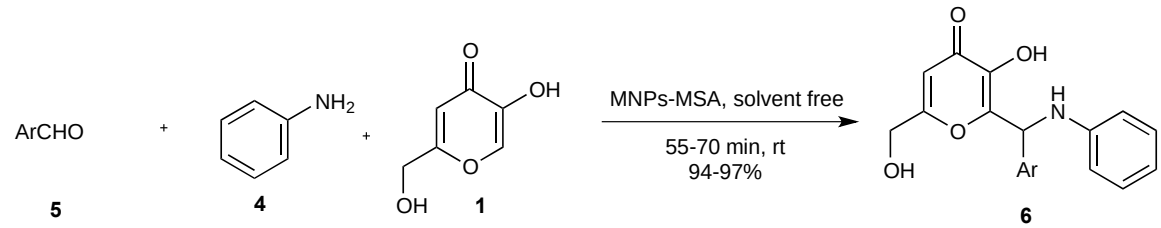


Figure 5. Title Words Growth occurrences by year in "kojic acid"

2. THE SYNTHESIS OF KOJIC ACID DERIVATIVES THROUGH DIFFERENT REACTIONS

2.1 2-Substituted aryl(amino)kojic acid derivatives

Magnetic nanoparticle-supported molybdate sulfuric acid (MNPs-MSA) was synthesized to be used as a catalyst in the one-pot synthesis of the 2-substituted aryl(amino)kojic acid scaffolds **6** from various aldehydes **5**, aniline **4**, and kojic acid **1** at room temperature without using solvent as shown in Scheme 1.[36]



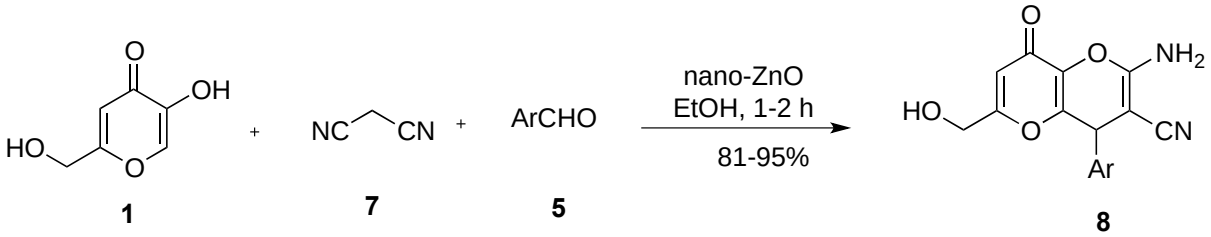
Ar = C_6H_5, 4-$MeOC_6H_5$, 4-ClC_6H_5, 4-BrC_6H_5, 2-naphtyl, 4-HOC_6H_5, 2-ClC_6H_5, 2-BrC_6H_5, 2-$OMeC_6H_5$, 4-MeC_6H_5

Scheme 1. The synthesis of 2-substituted aryl(amino)kojic acid scaffolds **6**

2.2. Dihydropyranopyran derivatives

The dihydropyranopyran derivatives **8** as the corresponding compounds were programmed through three-

component reactions by kojic acid **1**, different aromatic aldehydes **5,** and malononitrile **7** in the presence of the nano-ZnO as catalyst (Scheme 2).[37] Furthermore, this process was accomplished in different conditions *via* various catalysts as shown in Table 1.



1 **7** **5** **8**

Ar = C_6H_5, 4-BrC_6H_4, 3-$O_2NC_6H_4$, 3,4-$(OCH_2O)C_6H_3$, 1-naphthyl, thiophen-2-yl

Scheme 2. The synthesis of the dihydropyranopyran structures **8**

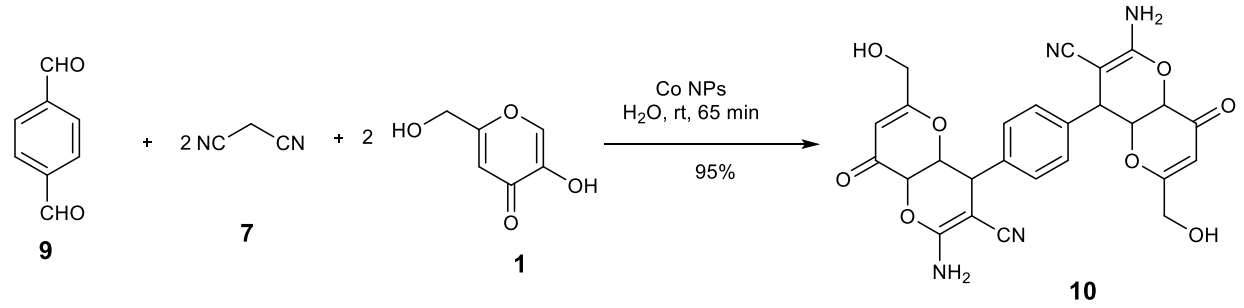
Table 1. The synthesis of the dihydro pyranopyran structures **8** through different catalysts

Entry	Solvent	Catalyst	Temp. (°C)	Time (min)	Yield (%)	Ref.
1	EtOH	nano-ZnO	reflux	1-2 h	81-94	37
2	H_2O	*β*-cyclodextrin	70	1 h.	83-96	38
3	EtOH	Zn(*L*-proline)$_2$	reflux	0.5-6 h	88-92	39
4	H_2O	Co NPs	rt	60-100	70-95	40
6	solvent-free	(SB-DBU)Cl[1]	rt	3-7	89-98	41
7	EtOH/H_2O	Fe_3O_4@SiO_2-IL-FC	rt	10-15	81-96	42
9	H_2O	MCM-41-SO_3H	90	35-50	87-95	43
10	H_2O	Cu_2O-CP	rt	1 h	85-92	44
11	H_2O	Fe_3O_4@SiO_2-*s*-triazinium	100	20-45	85-98	45

[1]1,8-Diazabicyclo[5.4.0]undec-7-ene immobilized on silica

2.3. *Bis*-2-aminodihydropyrano[3,2-*b*]pyran-3-carbonitrile derivatives

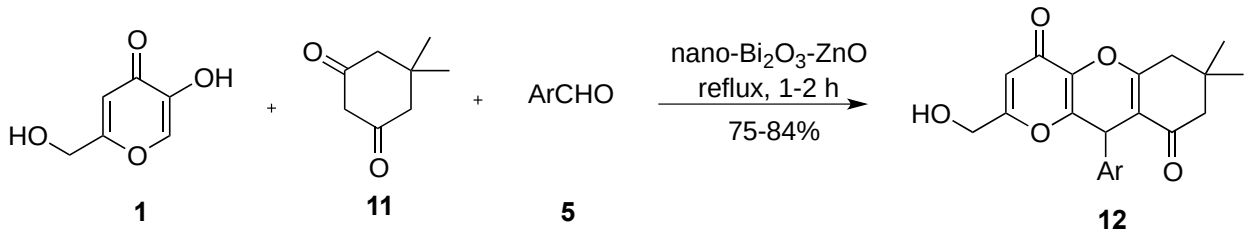
In this process, terephthalaldehyde **9** was applied to treat with malononitrile **7** and kojic acid **1** in the presence of the cobalt nanoparticles to yield the corresponding *bis*-2-aminodihydropyrano[3,2-*b*]pyran-3-carbonitrile **10** as the target compounds (Scheme 3).[40]



Scheme 3. The synthesis of the *bis*-2-aminodihydropyrano[3,2-*b*]pyran-3-carbonitrile derivatives **10**

2.4. Pyranochromene derivatives

The synthesis of pyranochromene structures **12** was developed through the three-component reactions of kojic acid **1**, different aromatic aldehydes **5**, and dimedone **11** by the nano-Bi_2O_3-ZnO as the catalyst as shown in Scheme 4. The catalyst Bi_2O_3-ZnO was synthesized by a sol-gel method by Bi^{3+} which was supported on ZnO nanoparticles to yield Bi_2O_3[26] as heterogeneous catalysis with metal oxides.[27,28,37] There are other reports related to this method under different conditions, as illustrated in Table 2.



Ar = C_6H_5, 4-ClC_6H_4, 2-ClC_6H_4, 3-$O_2NC_6H_4$, 4-$O_2NC_6H_4$, 2,4-$Cl_2C_6H_3$, 4-$MeOC_6H_4$, 4-MeC_6H_4

Scheme 4. The synthesis of pyranochromene structures **12**

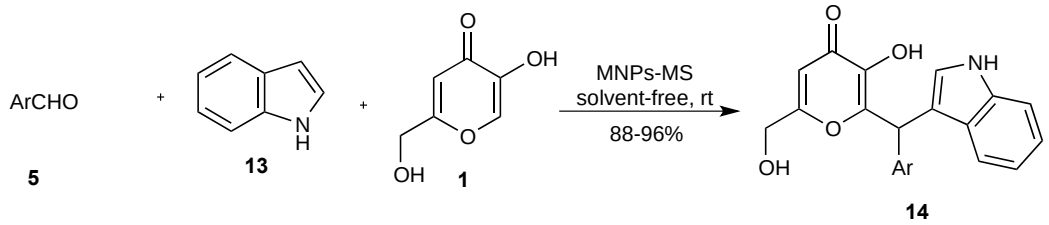
Table 2. The synthesis of pyranochromene structures **12** under different conditions

Entry	Solvent	Catalyst	Temp. (°C)	Time (min)	Yield (%)	Ref.
1	solvent-free	nano-Bi_2O_3-ZnO	reflux	1-2 h	75-84	[37]
2	solvent-free	PMAA-Fe_3O_4	110	10-25	64-96	[46]
3	H_2O	*β*-cyclodextrin	100	2 h	73-85	[38]
4	solvent-free	[SiPrPy]$AlCl_4$@MNPs	110	20-40	85-95	[47]

2.5. 2-Substituted aryl(indolyl)kojic acid derivatives

Magnetic nanoparticle-supported molybdate sulfuric acid (MNPs-MSA) was synthesized to be used as catalyst in the one-pot synthesis of the 2-substituted aryl(indolyl)kojic acid scaffolds **14** as the target compounds from various aldehydes **5**, indole **13** and kojic acid **1** at room temperature under the solvent-

free condition as shown in Scheme 5.[36] This method was also accomplished in the presence of the different catalysts such as nano SiO_2-OSO_3H, FAU-Zeolite, and $InCl_3$ through various conditions which were illustrated in Table 3.[24]



Ar = C_6H_5, 4-$MeOC_6H_5$, 4-ClC_6H_5,4-BrC_6H_5, 2-naphtyl, 4-HOC_6H_5, 2-ClC_6H_5, Et, 2-BrC_6H_5

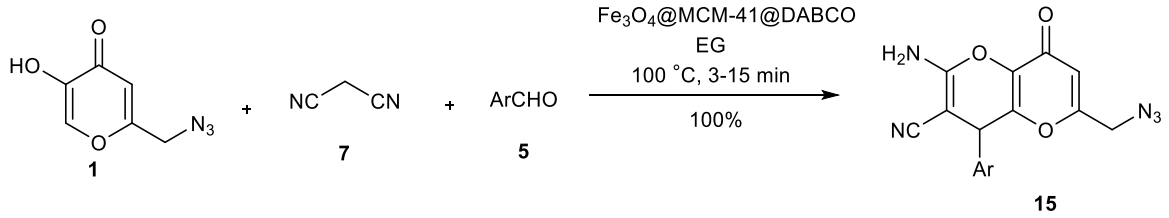
Scheme 5. The synthesis of 2-substituted aryl(indolyl)kojic acid scaffolds **14**

Table 3. The synthesis of the 2-substituted aryl(indolyl)kojic acid derivatives **14** under different conditions

Entry	Solvent	Catalyst	Temp. (°C)	Time (min)	Yield (%)	Ref.
1	solvent-free	MNPs-MS	rt	37-57	88-96	[36]
2	EtOH	nano SiO_2-OSO_3H	reflux	40-75	80-98	[48]
3	solvent-free	FAU-Zeolite	110	45-85	82-97	[49]
4	solvent-free	$InCl_3$	120	55-85	75-90	[50]

2.6. 2-Aminodihydropyrano[3,2-*b*]pyran-3-cyano derivatives

In this study, azido-KA **1** was treated with malononitrile **7** and different aldehydes **5** to yield 2-aminodihydropyrano[3,2-*b*]pyran-3-cyano cores **15** by using Fe_3O_4@MCM-41@DABCO as a catalyst at a suitable temperature. It is important to know that the synthesis of 2-aminodihydropyrano[3,2-*b*]pyran-3-cyano derivatives **15**, was accomplished using the magnetic nano-mesoporous Fe_3O_4@MCM-41@DABCO as catalyst in EG as a solvent. Also, this process was examined through different catalysts such as NH_4Cl, Fe_3O_4, and Fe_3O_4@MCM-41@DABCO which the last one gained high yield (Scheme 6).[51]



Ar = C_6H_5, 4-MeC_6H_4, 4-ClC_6H_4, 4-$O_2NC_6H_4$, 2-ClC_6H_4, 4-$MeOC_6H_4$, 4-HOC_6H_4, 3-HOC_6H_4, 2-$O_2NC_6H_4$, 2-HO-5-BrC_6H_3, 4-FC_6H_5, 2-$MeOC_6H_4$, 3-$O_2NC_6H_4$, 3,4,5-$MeOC_6H_2$, 2,4-ClC_6H_3

Scheme 6. The synthesis of the 2-aminodihydropyrano[3,2-*b*]pyran-3-cyano derivatives **15**

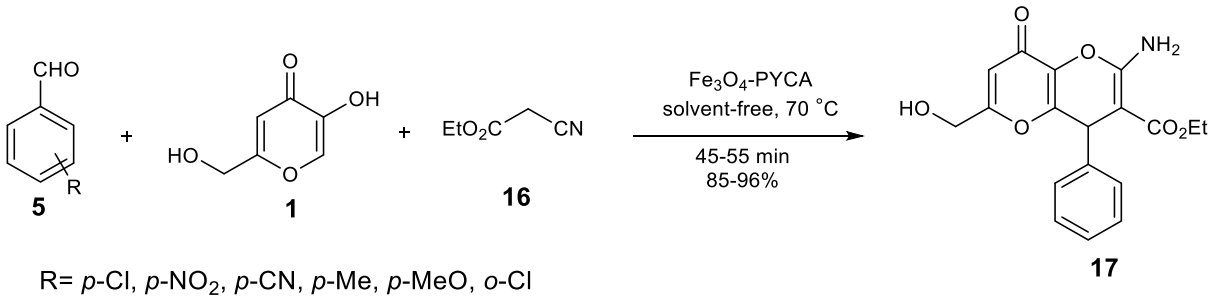
In this method, DABCO (1,4-diazabicyclo[2.2.2]octane)-modified magnetite was treated with the silica-MCM-41 shell to provide Fe_3O_4@silica-MCM-41@DABCO, which is used for the synthesis of 2-aminodihydropyrano[3,2-*b*]-pyran-3-cyano derivatives **37** as the corresponding compounds.[52] To obtain 2-aminodihydropyrano[3,2-*b*]pyran-3-cyano derivatives **15,** different conditions were used as shown in Table 4.[51]

Table 4. The synthesis of the 2-aminodihydropyrano[3,2-*b*]pyran-3-cyano derivatives **15** under different conditions

Entry	*Solvent*	*Catalyst*	Temp. (°C)	Time (min)	Yield (%)	Ref
1	EG	catalyst-free	100	12 h	trace	[51]
2	EG	Fe_3O_4	100	12 h	15	[51]
3	EG	Fe_3O_4@silica-MCM-41@DABCO	100	3-15	99	[51]
4	EtOH	NH_4Cl	78	12-24 h	90-99	[52]

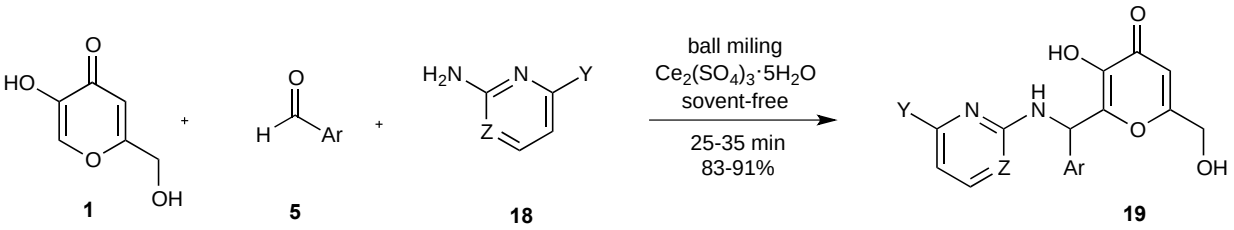
2.7. MISCELLANEOUS DERIVATIVES

In another work, Asghari and co-workers designed the novel catalyst by pyridine-4-carboxylic acid (PYCA) functionalized Fe_3O_4 nanoparticles as a magnetic hybrid heterogeneous catalyst to provide pyrano[3,2-*b*]pyranones **17** through the one-pot three-component reactions by various aromatic aldehydes **5**, kojic acid **1**, and ethyl cyanoacetate **16** under solvent-free conditions. The pros of this method were to be benign, simple by high yields and the catalyst was readily separated by the external magnet (Scheme 7).[53]

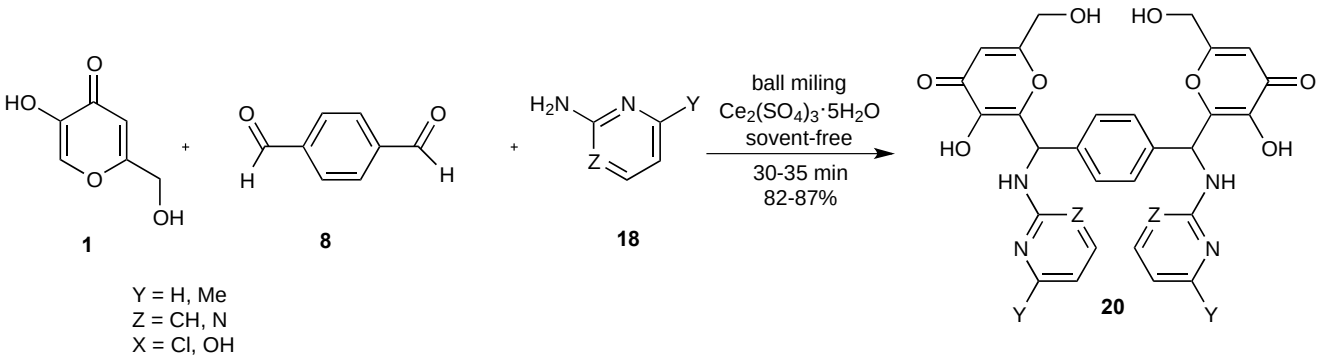


Scheme 7.The synthesis of the pyrano[3,2-*b*]pyranone structures **17**

In 2015, Teimuri-Mofrad and co-workers designed the process to obtain aminokojic acids **19**, **20**, respectively, through one-pot multicomponent reaction, by the reaction of kojic acid **1**, various aromatic aldehydes **5** or *bis*-aldehyde **8** and different heteroaromatic amines **18** in the presence of the cerium(III) sulfate as catalyst under the ball-milling condition at room temperature *via* solvent-free conditions (Schemes 8, 9).[54]



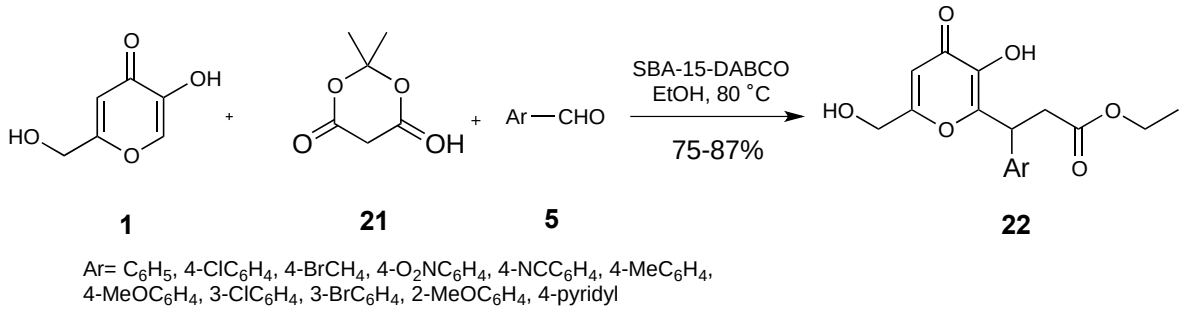
Scheme 8. The synthesis of aminokojic acids **19**



Scheme 9. The synthesis of aminokojic acids **20**

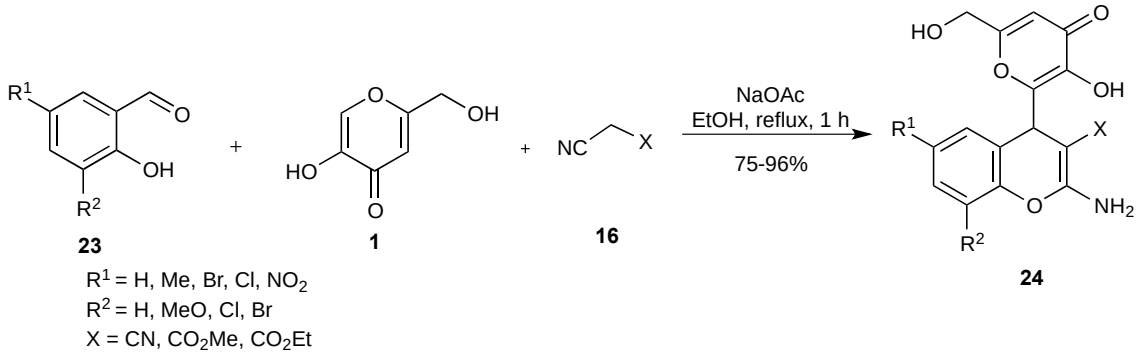
Novel kojic acid derivatives **22** were synthesized through a three-component condensation reaction as shown in Scheme 10. In this process, different aromatic aldehydes **5** reacted with Meldrum's acid **21**, and kojic acid **1**, in EtOH using DABCO-functionalized mesoporous SBA-15 as a catalyst. To obtain the

heterogeneous nonporous, solid-base catalyst, 1,4-diazabicyclo[2.2.2]octane was immobilized on mesoporous SBA-15 to give SBA-15-DABCO. The products were evaluated for their antioxidant activity *via* 1,1-diphenyl-2-picrylhydrazyl radical-scavenging compound.[55]



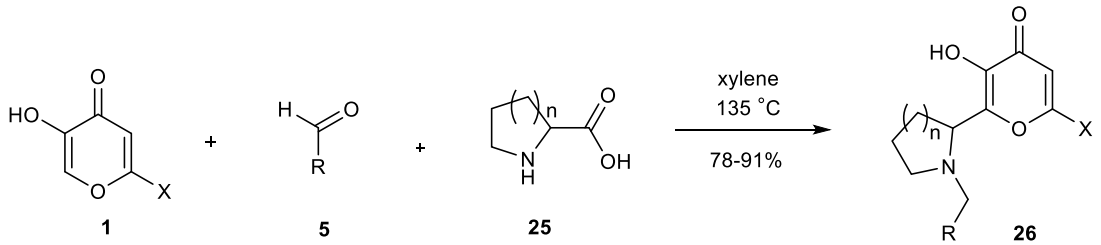
Scheme 10. The synthesis of novel kojic acid derivatives **22**

Multi-component assembling of various salicylaldehydes **23**, kojic acid **1**, and different substituted malononitriles **16** was disclosed by Elinson and co-workers in the presence of the sodium acetate, as a catalyst in EtOH to provide the substituted 2-amino-4-[3-hydroxy-6-(hydroxymethyl)-4-oxo-(4*H*)-pyran-2-yl]-(4*H*)-chromene-3-carbonitriles or -3-carboxylates **24** as products in 75–96% yields (Scheme 11).[56]



Scheme 11. The synthesis of 2-amino-4-[3-hydroxy-6-(hydroxymethyl)-4-oxo-(4*H*)-pyran-2-yl]-(4*H*)-chromene-3-carbonitriles or -3-carboxylates **24**

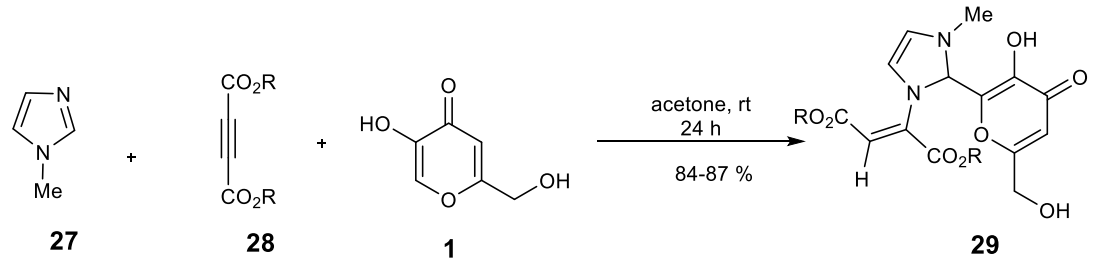
In this process, a novel class of kojic acid derivatives **1** was treated with the ***α***-amino acid **25**, and different aldehydes **5** through a three-component decarboxylative coupling reaction to provide 2-pyrrolidinyl and 2-piperidinyl substituted kojic acids **26** under thermal conditions as shown in Scheme 12.[57]



n= 1,2
R= *p*-Cl, *p*-F, *o*-F, *o*-Cl, *p*-NO_2, *p*-Br, *m*-Cl, *m*-Br, *m*-F
X= Me, CH_2OTBS

Scheme 12. The synthesis of 2-pyrrolidinyl-and 2-piperidinylkojic acid derivatives **26**

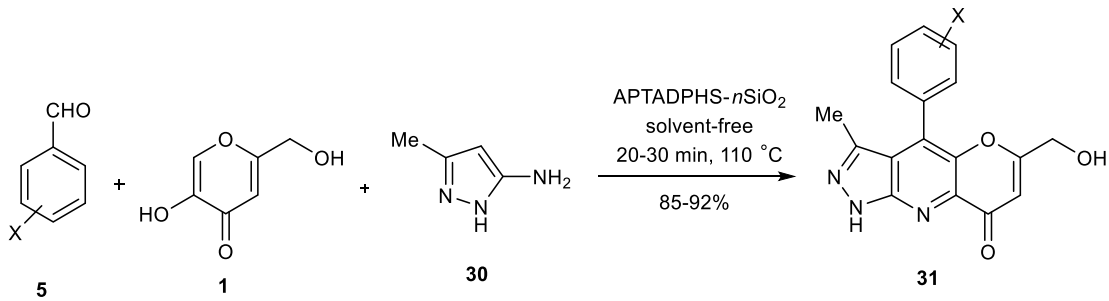
Pahlavan and co-workers reported the three-component reaction by *N*-methylimidazole **27**, variously activated acetylenes **28** and kojic acid **1** to afford 2,3-dihydroimidazoles **29** as shown in Scheme 13.[58]



R= Me, Et, *t*-Bu

Scheme 13. The synthesis of 2,3-dihydroimidazole derivatives **29**

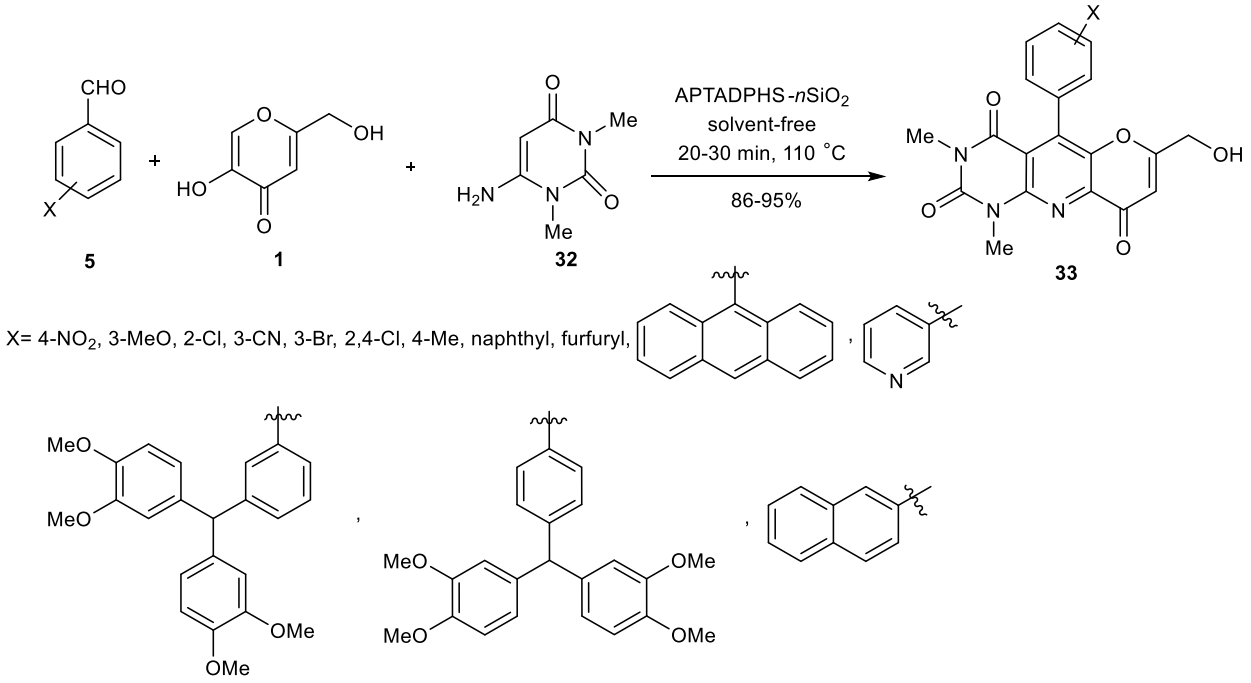
Rahman and co-workers developed the new approach through a one-pot three-component reaction of 3-methyl-1*H*-pyrazol-5-amine **30**, various aldehydes **5** and kojic acid **1** to provide fused pyridines **31** (Scheme 14). In this process**,** the triazine diphosphonium hydrogen sulfate ionic liquid used was supported on nano-silica to produce the catalyst APTDPHS-*n*SiO_2 to yield fused pyridine structures.[59]



X= 4-NO_2, 3-MeO, 2Cl

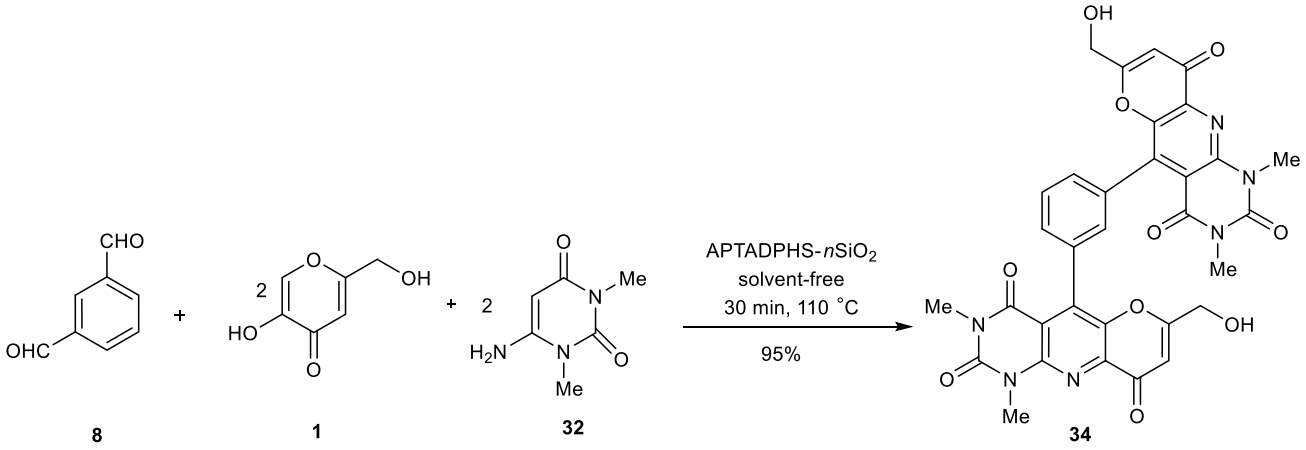
Scheme 14. The synthesis of fused pyridines derivatives **31**

In another attempt, the synthesis of the fused pyridines **33** was accomplished through the three-component reaction of various aldehydes **5**, kojic acid **1**, and 6-amino-1,3-dimethyluracil **32** using the ionic liquid aminopropyl-1,3,5-triazine-2,4-diphosphonium hydrogen sulfate supported on nano-silica, as catalyst without using solvent as shown in Scheme 15.[59]

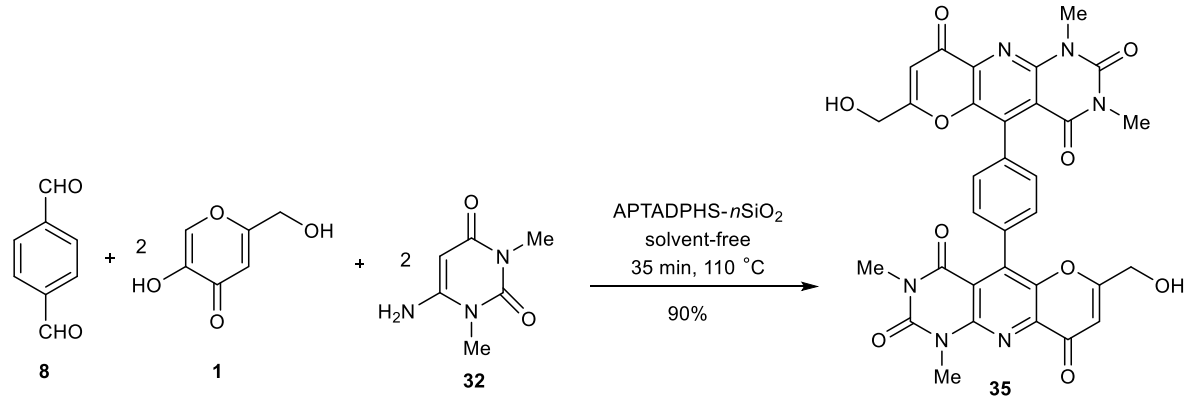


Scheme 15. The synthesis of fused pyridine derivatives **33**

The APTADPHS-nSiO$_2$ catalyst was examined for the synthesis of *bis*-annulated pyridine derivatives **34**, **35** respectively from terephthaldialdehyde or isophthaldialdehyde as *bis*-aldehydes **11**, kojic acid **1** and 6-amino-1,3-dimethyluracil **32** to produce the *bis*-pyridines **34** and **35** in acceptable yields under solvent-free conditions (Schemes 16, 17).[59]

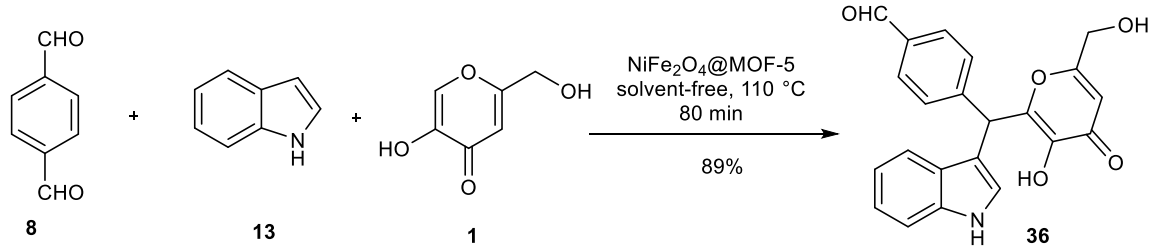


Scheme 16. The synthesis of *bis*-pyridine derivative **34**

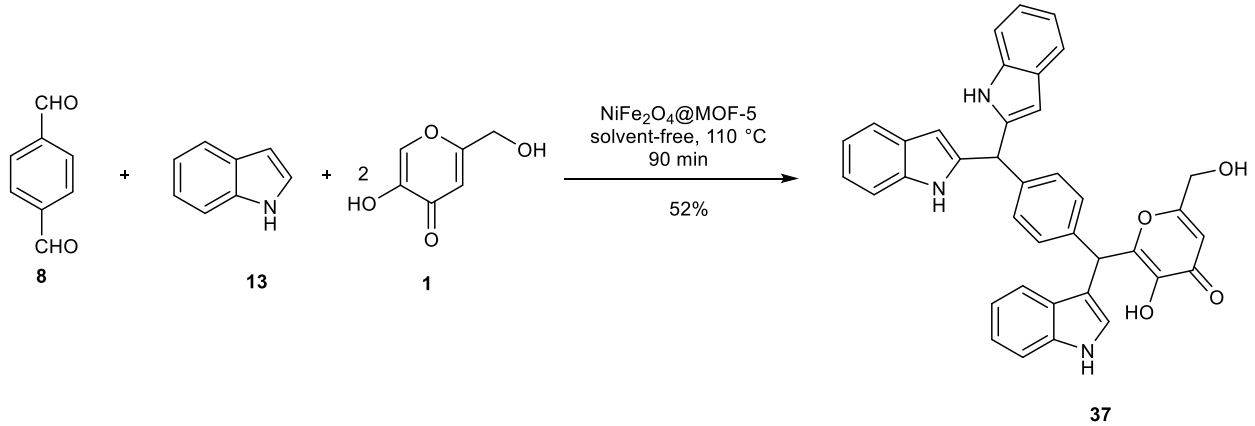


Scheme 17. The synthesis of *bis*-pyridine derivative **35**

A novel magnetic metal-organic structured $NiFe_2O_4$@MOF-5 was applied by Zhang and co-workers to yield 2-substituted aryl(indolyl)methylkojic acid derivatives **36**, **37**. In this process, 2-substituted aryl(indolyl)methylkojic acid cores **36** and **37** were provided through one-pot, the three-component reaction from dialdehydes **8**, indole **13** and kojic acid **1** (1 mmol) (Scheme 19) or (2 mmol) (Scheme 20) under solvent-free conditions respectively.[60]



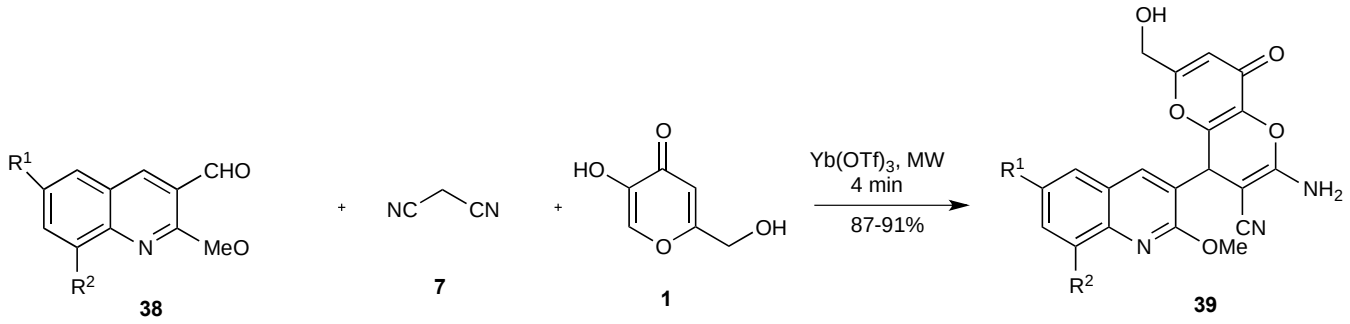
Scheme 18. The synthesis of 2-substituted aryl(indolyl)methylkojic acid derivative **36**



Scheme 19. The synthesis of 2-substituted aryl(indolyl)methylkojic acid derivative **37**

A series of 2-methoxy-3-(2-amino-3-cyano-6-(hydroxymethyl)-8-oxo-4,8-dihydropyrano[3,2-*b*]pyran-4-yl)quinolone **39** as corresponding compounds were synthesized by Kumarasamy and co-workers in 2019.

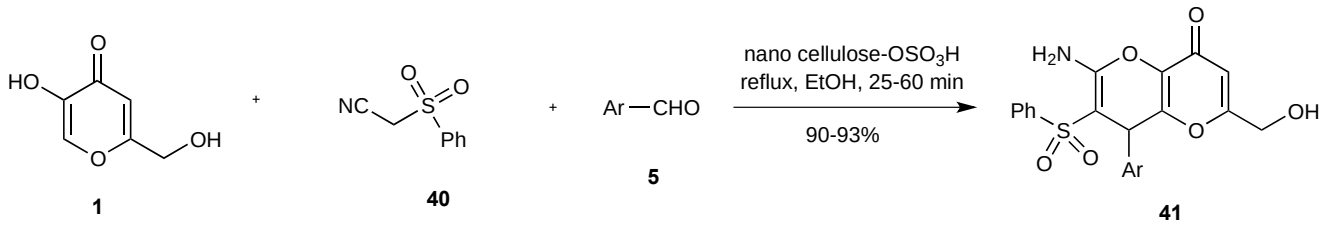
In this study, various 2-methoxy-3-formylquinolones **38** were treated with malononitrile **7**, and kojic acid **1** to furnish the product **39** in the presence of the metal triflates $Yb(OTf)_3$ through one-pot, multi-component reaction under microwave conditions (Scheme 20).[61]



R^1 = H, Me, MeO
R^2 = H, Me

Scheme 20. The synthesis of the 2-methoxy-3-(2-amino-3-cyano-6-(hydroxymethyl)-8-oxo-4,8-dihydropyrano[3,2-*b*]pyran-4-yl)quinolone derivatives **39**

The target compounds 6-amino-2-(hydroxymethyl)-8-aryl-7-(phenylsulfonyl)pyrano[3,2-*b*]pyran-4(8*H*)-ones **41** as pyran-annulated heterocyclic derivatives were synthesized through multi-component reaction. In this process, different aromatic aldehydes **5** reacted with kojic acid **1** and phenylsulfonylacetonitrile **40** using nano-cellulose-OSO_3H as catalyst to yield the target compounds **41** (Scheme 21).[62]



Ar= 3-$O_2NC_6H_4$, 2-HO-5-$O_2NC_6H_3$, 4-Cl-3-$O_2NC_6H_3$, 4-NCC_6H_4, 3-formylchromone, 2-HO-3-$MeOC_6H_3$

Scheme 21. The synthesis of the 6-amino-2-(hydroxymethyl)-8-aryl-7-(phenylsulfonyl)-pyrano[3,2-*b*]pyran-4(8*H*)-one derivatives **41**

3. CONCLUSIONS

In conclusion, using kojic acid conclusively established a wide-ranging of compounds with high biological activities. The presence of this compound in pharmaceutical applications shows the importance

of the different releasing scaffolds through different conditions. Also, in this review the importance of the kojic acid was studied through biobliometric appraoches.

ABBREVIATION

List of Abbreviations			
Abbreviate	**Full name**	**Abbreviate**	**Full name**
KA	kojic acid	EG	ethylene glycol
FAU-Zeolite	faujasite	$Yb(OTf)_3$	ytterbium(III) triflates
$ClSO_3H$	chlorosulfonic acid	PYCA	pyridine-4-carboxylic acid
DABCO	1,4-diazabicyclo[2.2.2.]octane	APTDPHS-*n*SiO_2	triazine diphosphonium hydrogen sulfate ionic liquid- nano-silica
MNPs-MSA	magnetic nanoparticle-supported molybdate sulfuric acid		

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Fatemeh Mohajer was born in Tehran, Iran and she received her B.Sc. in Applied Chemistry from Bu-Ali Sina University and M.Sc degree in Organic Chemistry from Azad University in Karaj. She is Ph.D student under the supervision of Prof. Ghodsi Mohammadi Ziarani in Alzahra University in Tehran, Iran.

Ghodsi Mohammadi Ziarani received her B.Sc. degree in Chemistry from Teacher Training University, Tehran, Iran, in 1987, her M.Sc. degree in Organic Chemistry from the Teacher Training University, Tehran, Iran, under the supervision of Professor Jafar Asgarin and Professor Mohammad Ali Bigdeli in 1991 and her Ph.D. degree in asymmetric synthesis (Biotransformation) from Laval University, Quebec, Canada under the supervision of Professor Chenevert, in 2000. She is Full Professor of Organic Chemistry in the chemistry department of Alzahra University. Her research interests include organic synthesis, heterocyclic synthesis, asymmetric synthesis, natural products synthesis, synthetic methodology and applications of nano-heterogeneous catalysts in multicomponent reactions.