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A solvent-free protocol for the green synthesis of heterocyclic chalcones

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ABSTRACT

Twenty chalcones were prepared by grinding equimolar quantities of (hetero) aryl methyl ketone with (hetero) aryl aldehyde in presence of sodium hydroxide in solvent free condition. Such an attempt has not yet been made or available for the synthesis of heterocyclic chalcones mentioned in this work. The synthesis was found to be simple, efficient in terms of short reaction time, excellent yields and afford single product as indicated in TLC. The synthesized compounds were characterized by means of their IR, ^{1}H NMR spectral data and elemental analysis. This method may be a promising alternative to the conventional methods.

Keywords: grinding, aldehyde, ketone, chalcone, green synthesis

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INTRODUCTION

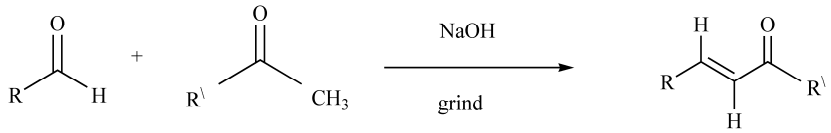
The term Green chemistry means the design and development of chemical process to reduce or eliminate the use and generation of chemicals hazardous to the environment. Solvents used in organic reactions are often toxic, expensive and disposal of such solvents is a serious threat to nature .Thus, design of solvent free organic synthesis has received tremendous attention in recent years. [1]

Chalcones are aromatic ketones that form the central core for a variety of important biological compounds. The highly electrophilic three carbon α,β-unsaturated carbonyl system in chalcone has assumed importance because of their versatility in the synthesis of many heterocyclics and also the compounds with the backbone of chalcone have been reported to possess various biological activities like anti-inflammatory, anti-ulcerative, analgesic, anti-viral, anti-fungal, anti-malarial, anti-bacterial and anticancer activities.[2-9] Chalcone bears a very good synthon for the synthesis of variety of heterocyclic compounds like thiazine, oxazine, isoxazole, pyrazole, diazepine, pyridine, pyrimidine. [10-11] .Therefore, the synthesis of chalcones continues to attract much interest in organic chemistry.

Several methods have been reported for the synthesis of chalcones. The most commonly used method is the base-catalyzed Claisen Schmidt reaction of a methyl ketone and aldehyde using sodium hydroxide (NaOH),[12] potassium hydroxide (KOH),[13] barium hydroxide $Ba(OH)_2$[14] and lithium hydroxide ($LiOH\cdot H_2O$) [15]. The acid-catalyzed synthesis of chalcones using aluminum trichloride ($AlCl_3$),[16] dry HCl,[17] boron trifluoride-etherate (BF_3-Et_2O),[18] titanium tetrachloride ($TiCl_4$) [19] and ruthenium trichloride ($RuCl_3$) [20] has also been reported. Reaction is carried out under acid-catalyzed conditions, especially when one of the partners, either the aldehyde or the acetophenone, is base sensitive. [21, 22]

Solvent free or solid state reaction may be carried out using the reactants alone or incorporating them in clays, zeolites, silica, alumina or other matrices [23]. Herein, we report a simple, atom efficient and eco-friendly method

for the synthesis of chalcones containing at least one heterocyclic ring. Twenty chalcones were prepared by grinding equimolar quantities of (hetero) aryl methyl ketone with (hetero) aryl aldehyde in presence of sodium hydroxide (scheme 1). To the best of our knowledge, such an attempt has not yet been made or available for the synthesis of heterocyclic chalcones mentioned in this work. All the twenty compounds reported here were prepared in the absence of solvent and the reaction found to be simple, efficient in terms of short reaction time, excellent yields and afford single product as indicated in TLC.



scheme 1

MATERIALS AND METHODS

Experimental

All the reagents and solvents used were of synthetic grade .The melting points were recorded in open capillary method and are uncorrected. IR spectra were recorded using JASCO FT-IR spectrophotometer. ^{1}H NMR spectrum was recorded using $CDCl_3$ on Bruker Avance (400 MHz) and their chemical shifts are recorded in δ (parts per million) units with respect to tetramethyl silane (TMS) as internal standard. Elemental analysis was performed on vario MICRO V2.0.3,Elementar analysis system GmbH. Progress of the reactions was monitored using TLC, performed on precoated silica gel-60 F_{254} (Merck) plates using hexane-ethyl acetate (2:1, v/v) as solvent system.

General procedure for the preparation of (1-20)

A mixture of appropriate aldehyde (4 mmol) and methylketone (4 mmol) was mixed thoroughly then sodium hydroxide (4 mmol) was added and ground with a pestle in an open mortar at room temperature for the time mentioned in table 1. The mixture solidifies and the solid broke up into small particles, the completion of the reaction was monitored by TLC .The product formed was washed with water to remove the traces of sodium hydroxide to give the corresponding chalcone.

Table 1Solvent free Synthesis of chalcones (1-20)

Entry	Aldehyde (R)	Ketone (R\ )	Reaction time (min)	Yield (%)	Mp (^{0}C)
1	2-thienyl	2-thienyl	6-8	93	98-100
2	2-thienyl	4-chloro acetophenone	5-7	95	118-120
3	4-ClC_6H_4	2-thienyl	5-7	94	128-130
4	2-thienyl	4-fluro acetophenone	3-6	93	72-74
5	2-thienyl	4-methyl acetophenone	3-6	94	74-76
6	4-FC_6H_4	2-thienyl	2-5	95	130-132
7	2-furyl	2-acetyl furan	2-5	90	88-90
8	2-furyl	4-fluro acetophenone	2-6	90	74-76
9	2-furyl	4-chloro acetophenone	2-5	95	82-84
10	3,4,5-$(OCH_3)_3$ C_6H_2	2-thienyl	2-5	91	165-166
11	3-$NO_2C_6H_4$	2-thienyl	4-6	91	80-82
12	4-FC_6H_4	2-acetyl furan	3-6	93	120-122
13	3,4,5-$(OCH_3)_3$ C_6H_2	2-acetyl furan	2-5	93	160-162
14	4-ClC_6H_4	2-acetyl furan	2-5	95	146-148
15	3,4,5-$(OCH_3)_3$ C_6H_2	2-acetyl-5-methylfuran	2-5	95	130-132
16	2-furyl	2-acetyl-5-methylfuran	3-6	93	88-90
17	3-$NO_2C_6H_4$	2-acetyl-5-methylfuran	2-5	93	150-152
18	2-furyl	4-bromo acetophenone	2-5	95	86-88
19	2-thienyl	4-bromo acetophenone	2-5	95	136-138
20	4-FC_6H_4	2-acetyl pyridine	5-7	95	90-92

RESULTS AND DISCUSSION

Spectral and physical data of the synthesized compounds (1-20)

(E)-1,3-di(thiophen-2-yl)prop-2-en-1-one (1)

Pale yellow solid compound, Yield 93%; m.p.: 98-100 ^{0}C; R_f :0.62; ^{1}H NMR (400 MHz, $CDCl_3$) 7.21 (d, 1H, J = 15.29 Hz, -CO-CH=),7.97 (d, 1H, J = 15.24 Hz, =CH-Ar), 7.0-7.8 (6H, Ar-H), IR (KBr, cm^{-1}): 3093 (Ar-C-H str), 1637 (C=O str), 1572 (C=C str), 973 (CH=CH trans). Anal. calcd. for $C_{11}H_8OS_2$: C, 59.97; H, 3.66; S, 29.11. Found: C, 59.61; H, 3.78; N, 29.42.

(E)-1-(4-chlorophenyl)-3-(thiophen-2-yl)prop-2-en-1-one (2)
Yellow solid compound, Yield 95%; m.p.: 118-120 ^{0}C; R_f : 0.69; ^{1}H NMR (400 MHz, $CDCl_3$) 7.27 (d, 1H, *J* = 15.29 Hz, -CO-CH=), 7.94 (d, 1H, *J* = 15.32 Hz, =CH-Ar), 7.08-7.96 (7H, Ar-H). IR (KBr, cm^{-1}): 3083 (Ar-C-H str), 1650 (C=O str), 1590 (C=C str), 972 (CH=CH trans), 678 (C-Cl str) . Anal. calcd. for $C_{13}H_9ClOS$: C, 62.78; H, 3.65; S, 12.89 . Found: C, 62.63; H, 3.31; S, 12.73.

(E)-3-(4-chlorophenyl)-1-(thiophen-2-yl)prop-2-en-1-one (3)
Colourless solid compound, Yield 94%; m.p.: 128-130 ^{0}C; R_f : 0.72; ^{1}H NMR (400 MHz, $CDCl_3$) 7.39 (d, 1H, *J* = 15.56 Hz, -CO-CH=), 7.79 (d, 1H, *J* = 15.52 Hz,=CH-Ar), 7.1-7.87 (7H, Ar-H), IR (KBr, cm^{-1}): 3071 (Ar-C-H str), 1649 (C=O str), 1599 (C=C str), 989 (CH=CH trans), 680 (C-Cl str) . Anal. calcd. for $C_{13}H_9ClOS$: C, 62.78; H, 3.65; S, 12.89. Found: C, 62.82; H, 3.52; S, 12.64.

(E)-1-(4-fluorophenyl)-3-(thiophen-2-yl)prop-2-en-1-one (4)
Offwhite solid compound, Yield 94%; m.p.: 72-74 ^{0}C; R_f : 0.71; ^{1}H NMR (400 MHz, $CDCl_3$) 7.30 (d, 1H, *J* = 15.28 Hz, -CO-CH=), 7.95 (d, 1H, *J* = 15.36 Hz, =CH-Ar), 7.0-8.0 (7H, Ar-H), IR (KBr, cm^{-1}): 3072 (Ar-C-H str), 1658 (C=O str), 1596 (C=C str), 972 (CH=CH trans). Anal. calcd. for $C_{13}H_9FOS$: C, 67.22; H, 3.91; S, 13.80. Found: C, 67.38; H, 3.62; S, 13.66.

(E)-3-(thiophen-2-yl)-1-(p-tolyl)prop-2-en-1-one (5)
Yellow solid compound, Yield 94%; m.p.: 74-76 ^{0}C; R_f : 0.72; ^{1}H NMR (400 MHz, $CDCl_3$) 2.4 (3H, -CH_3), 7.33 (d, 1H, *J* = 15.36 Hz, -CO-CH=), 7.93 (d, 1H, *J* = 15.38 Hz, =CH-Ar), 7.07-7.95 (7H, Ar-H), IR (KBr, cm^{-1}): 3024 (Ar-C-H str), 1656 (C=O str), 1594 (C=C str), 969 (CH=CH trans). Anal. calcd. for $C_{14}H_{12}OS$: C, 73.65; H, 5.30; S, 14.04. Found: C, 73.82; H, 5.66; S, 14.34.

(E)-3-(4-fluorophenyl)-1-(thiophen-2-yl)prop-2-en-1-one (6)
Yellow solid, Yield 95%; m.p.: 130-132 ^{0}C; R_f : 0.68; ^{1}H NMR (400 MHz, $CDCl_3$) 7.35 (d, 1H, *J* = 15.56 Hz,-CO-CH=), 7.81 (d, 1H, *J* = 15.6 Hz, =CH-Ar)7.08-7.87 (7H, Ar-H), IR (KBr, cm^{-1}): 3073 (Ar-C-H str), 1649 (C=O str), 1588 (C=C str), 980 (CH=CH trans). Anal. calcd. for $C_{13}H_9FOS$: C, 67.22; H, 3.91; S, 13.80. Found: C, 67.42; H, 3.67; S, 13.78.

(E)-1,3-di(furan-2-yl)prop-2-en-1-one (7)
Yellow solid, Yield 90%; m.p.: 88-90 ^{0}C; R_f : 0.55; ^{1}H NMR (400 MHz, $CDCl_3$) 7.32 (d, 1H, *J* = 15.49 Hz,-CO-CH=), 7.63 (d, 1H, *J* = 15.52 Hz, =CH-Ar), 6.51-7.53 (6H, Ar-H), IR (KBr, cm^{-1}): 3083 (Ar-C-H str), 1654 (C=O str), 1602 (C=C str), 971 (CH=CH trans). Anal. calcd. for $C_{11}H_8O_3$: C, 70.21; H, 4.29. Found: C, 70.46; H, 4.93; H,4.61.

(E)-1-(4-fluorophenyl)-3-(furan-2-yl)prop-2-en-1-one (8)
Pale yellow solid, Yield 90%; m.p.: 74-76 ^{0}C; R_f : 0.58; ^{1}H NMR (400 MHz, $CDCl_3$) 7.40 (d, 1H, *J* = 15.32 Hz,-CO-CH=), 7.59 (d, 1H, *J* = 15.28 Hz,=CH-Ar), 6.51-8.0 (7H, Ar-H), IR (KBr, cm^{-1}): 3065 (Ar-C-H str), 1663 (C=O str), 1593 (C=C str), 971 (CH=CH trans). Anal. calcd. for $C_{13}H_9FO_2$: C, 72.22; H, 4.20. Found: C, 72.22; H, 4.42.

(E)-1-(4-chlorophenyl)-3-(furan-2-yl)prop-2-en-1-one (9)
Pale yellow solid, Yield 95%; m.p.: 82-84 ^{0}C; R_f :0.65; ^{1}H NMR (400 MHz, $CDCl_3$) 7.3 (d, 1H, *J* = 15.32 Hz,-CO-CH=), 7.57(d, 1H, *J* = 15.28 Hz, =CH-Ar), 6.51-7.98 (7H, Ar-H), IR (KBr, cm^{-1}): 3059 (Ar-C-H str), 1655 (C=O str), 1597 (C=C str), 973 (CH=CH trans). Anal. calcd. for $C_{13}H_9ClO_2$: C, 67.11; H, 3.90. Found: C, 67.42; H, 3.76.

(E)-1-(thiophen-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (10)
Pale yellow solid, Yield 91%; m.p.: 165-166 ^{0}C; R_f : 0.50; ^{1}H NMR (400 MHz, $CDCl_3$) 7.31 (d, 1H, *J* = 15.56 Hz,-CO-CH=), 7.77 (d, 1H, *J* = 15.48 Hz, =CH-Ar), 7.1-7.89 (5H, Ar-H), IR (KBr, cm^{-1}): 3086 (Ar-C-H str), 1644 (C=O str), 1596 (C=C str), 976 (CH=CH trans). Anal. calcd. for $C_{16}H_{16}O_4S$: C, 63.14; H, 5.30; S, 10.54. Found: C, 63.42; H, 5.12; S, 10.76.

(E)-3-(3-nitrophenyl)-1-(thiophen-2-yl)prop-2-en-1-one (11)
White solid, Yield 91%; m.p.: 80-82 ^{0}C; R_f : 0.50; ^{1}H NMR (400 MHz, $CDCl_3$) 7.53(d, 1H, *J* = 15.56 Hz,-CO-CH=), 7.87 (d, 1H, *J* = 15.64 Hz, =CH-Ar), 7.2-8.52 (7H, Ar-H), IR (KBr, cm^{-1}): 3074 (Ar-C-H str), 1650 (C=O str), 1598 (C=C str), 977 (CH=CH trans). Anal. calcd. for $C_{13}H_9NO_3S$: C, 60.22; H, 3.50; N, 5.40; S, 12.37. Found: C, 60.41; H, 3.73; N, 5.36; S, 12.62.

(E)-3-(4-fluorophenyl)-1-(furan-2-yl)prop-2-en-1-one (12)
White solid, Yield 93%; m.p.: 120-122 ^{0}C; R_f : 0.79; ^{1}H NMR (400 MHz, $CDCl_3$) 7.38 (d, 1H, *J* = 15.76 Hz,-CO-CH=), 7.84 (d, 1H, *J* = 15.76 Hz, =CH-Ar), 6.59-7.67 (7H, Ar-H), IR (KBr, cm^{-1}): 3096 (Ar-C-H str), 1655 (C=O str), 1589 (C=C str), 972 (CH=CH trans). Anal. calcd. for $C_{13}H_9FO_2$: C, 72.22; H, 4.20. Found: C, 72.46; H, 4.38.

(E)-1-(furan-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (13)
Pale yellow solid, Yield 93%; m.p.: 160-162 ^{0}C; R_f :0.53; ^{1}H NMR (400 MHz, $CDCl_3$) 3.9(9H, -OCH_3), 7.34 (d, 1H, *J* = 15.64 Hz,-CO-CH=), 7.80 (d, 1H, *J* = 15.72 Hz, =CH-Ar), 6.6-7.6 (5H, Ar-H), IR (KBr, cm^{-1}): 3041 (Ar-C-H str), 1655 (C=O str), 1598 (C=C str), 975 (CH=CH trans). Anal. calcd. for $C_{16}H_{16}O_5$: C, 66.66; H, 5.59. Found: C, 66.81; H, 5.82.

(E)-3-(4-chlorophenyl)-1-(furan-2-yl)prop-2-en-1-one (14)
Colourless solid, Yield 95%; m.p.:146-148 ^{0}C; R_f :0.65; ^{1}H NMR (400 MHz, $CDCl_3$) 7.43 (d, 1H, *J* = 15.76 Hz,-CO-CH=), 7.82 (d, 1H, *J* = 15.8 Hz, =CH-Ar), 6.60-7.66 (7H, Ar-H), IR (KBr, cm^{-1}): 3059 (Ar-C-H str), 1665 (C=O str), 1598 (C=C str), 974 (CH=CH trans). Anal. calcd. for $C_{13}H_9ClO_2$: C, 67.11; H, 3.90. Found: C, 67.42; H, 3.88.

(E)-1-(5-methylfuran-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (15)
Yellow solid, Yield 95%; m.p.:130-132 ^{0}C; R_f : 0.31; ^{1}H NMR (400 MHz, $CDCl_3$) 2.45 (s, 3H, -CH_3), 3.90 (s,3H, -OCH_3),3.92 (s,6H, -OCH_3), 7.26 (d, 1H, *J* = 15.64 Hz,-CO-CH=), 7.78 (d, 1H, *J* = 15.64 Hz, =CH-Ar), 6.2-7.27 (4H, Ar-H), IR (KBr, cm^{-1}): 3071 (Ar-C-H str), 1652 (C=O str), 1596 (C=C str), 971 (CH=CH trans). Anal. calcd. for $C_{17}H_{18}O_5$: C, 67.54; H, 6.00. Found: C, 67.81; H, 6.32.

(E)-1-(5-methylfuran-2-yl)-3-(3-nitrophenyl)prop-2-en-1-one (16)
Colourless solid, Yield 93%; m.p.: 88-90 ^{0}C; R_f :0.68; ^{1}H NMR (400 MHz, $CDCl_3$,) 2.43 (s, 3H, -CH_3),7.26 (d, 1H, *J* = 15.44 Hz,-CO-CH=), 7.60 (d, 1H, *J* = 15.44 Hz, =CH-Ar), 6.2-7.52 (5H, Ar-H), IR (KBr, cm^{-1}): 2998 (Ar-C-H str), 1649 (C=O str), 1597 (C=C str), 970 (CH=CH trans). Anal. calcd. for $C_{12}H_{10}O_3$: C, 71.28; H, 4.98. Found: C, 71.14; H, 4.61.

(E)-1-(5-methylfuran-2-yl)-3-(3-nitrophenyl)prop-2-en-1-one (17)
Yellow solid, Yield 93%; m.p.: 150-152 ^{0}C; R_f : 0.51; ^{1}H NMR (400 MHz, $CDCl_3$) 2.47 (s, 3H, -CH_3),7.51 (d, 1H, *J* = 15.76 Hz,-CO-CH=), 7.86 (d, 1H, *J* = 15.8 Hz, =CH-Ar), 6.26-8.52 (6H, Ar-H), IR (KBr, cm^{-1}): 3078 (Ar-C-H str), 1655 (C=O str), 1596 (C=C str), 974 (CH=CH trans). Anal. calcd. for $C_{14}H_{11}NO_4$: C, 65.37; H, 4.31; N, 5.44. Found: C, 65.64; H, 4.68; N, 5.63.

(E)-1-(4-bromophenyl)-3-(furan-2-yl)prop-2-en-1-one (18)
Yellow solid, Yield 95%; m.p.: 86-88 ^{0}C; R_f : 0.66; ^{1}H NMR (400 MHz, $CDCl_3$) 7.40 (d, 1H, *J* = 15.28 Hz,-CO-CH=), 7.56 (d, 1H, *J* = 15.28 Hz, =CH-Ar), 6.52-7.91 (7H, Ar-H), IR (KBr, cm^{-1}): 3057 (Ar-C-H str), 1654 (C=O str), 1593 (C=C str), 972 (CH=CH trans). Anal. calcd. for $C_{13}H_9BrO_2$: C, 56.34; H, 3.27. Found: C, 56.18; H, 3.57.

(E)-1-(4-bromophenyl)-3-(thiophen-2-yl)prop-2-en-1-one (19)
Yellow solid, Yield 95%; m.p.: 136-138 ^{0}C; R_f : 0.78; ^{1}H NMR (400 MHz, $CDCl_3$) 7.27 (d, 1H, *J* = 15.28 Hz,-CO-CH=), 7.95 (d, 1H, *J* = 15.36 Hz, =CH-Ar), 7.09-7.87 (7H, Ar-H), IR (KBr, cm^{-1}): 3081 (Ar-C-H str), 1650 (C=O str), 1587 (C=C str), 972 (CH=CH trans). Anal. calcd. for $C_{13}H_9BrOS$: C, 53.26; H, 3.09; S, 10.94. Found: C, 53.18; H, 3.36; S, 10.74.

(E)-3-(4-fluorophenyl)-1-(pyridin-2-yl)prop-2-en-1-one (20)
Yellow solid, Yield 95%; m.p.: 90-92 ^{0}C; R_f : 0.36; ^{1}H NMR (400 MHz, $CDCl_3$) 7.1 (d, 1H, *J* = 15.76 Hz,-CO-CH=), 8.22 (d, 1H, *J* = 16.12 Hz, =CH-Ar), 7.0-8.75 (8H, Ar-H), IR (KBr, cm^{-1}): 3065 (Ar-C-H str), 1655 (C=O str), 1588 (C=C str), 973 (CH=CH trans). Anal. calcd. for $C_{14}H_{10}FNO$: C, 74.00; H, 4.44; N, 6.16. Found: C, 74.36; H, 4.16; N, 6.31.

DISCUSSION

The conventional synthesis of chalcones from aldehyde and methyl ketone involves dissolving the ketone in a basic alcoholic solution and this is subsequently added to an alcoholic solution of the aldehyde, resulting in the formation of the product as a precipitate which is then washed with water and recrystallized from ethanol. We have found that grinding the reactants together in the presence of catalytic amount of sodium hydroxide, the aldol condensation proceeds as in solution by aggregating the reaction mixture using a mortar and pestle. Aldehyde and ketone upon the addition of NaOH immediately turns yellow indicating the formation of enolate , upon gentle grinding the viscosity

rapidly increases and forms solid after 2-7 min. It was observed that vigorous grinding leads to the formation of some unwanted products which was indicated in TLC. The pure product was isolated in quantitative yield by washing with water to remove excess of base, the reaction furnished chalcones (1-20) in 90-95% yield. It seems that the presence of electron releasing and withdrawing substituent has no significant effect in the formation of products in the reported synthesis. All the reactions were proceeded in the same manner and there was no significant difference in yield, purity and reaction time when different substituent were present on aldehyde and ketone. All The product obtained were characterized on the basis of their analytical and spectral data.IR spectra of compounds exhibited absorptions at 3000-3100 cm^{-1} for (aromatic C-H stretching), 1637-1655 cm^{-1} (C=O stretching),1572-1597 cm^{-1} (C=C stretching), 970-980 cm^{-1} (-CH=CH- trans). In the ^1H NMR ($CDCl_3$) spectra of the chalcones, the protons of α-β unsaturated system absorbed as two doublets with coupling constant J = 15-16 Hz, it confirms that the double bond of enone moiety is trans configured. The proton spectral data agree with respect to the number of protons and their chemical shifts with the proposed structures. Chemical composition of the compounds was identified using elemental analysis and the calculated values are very close to each other and are within the error limits (±0.4% of the calculated value).

In conclusion, the method shown here is the convenient, eco-friendly, economical and efficient method for the synthesis of heterocyclic chalcone and this method may be a promising alternative to the conventional methods.

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