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# Sustainable One-Pot Multi-Component Organic Synthesis (Biginelli Synthesis) Mediated by Fruit Juices as Bio-catalysts

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**Abstract:** *A sustainable and environmentally benign protocol for the Biginelli multi-component reaction has been developed using natural fruit juices as bio-catalysts. Fresh extracts of Karwand (*Carissa carandas*), Kokam (*Garcinia indica*), Mango (*Mangifera indica*), and Strawberry (*Fragaria × ananassa*) were evaluated for their catalytic efficiency in the one-pot condensation of aldehydes, β-ketoesters, and urea/thiourea for the synthesis of dihydropyrimidinones (DHPMs). The methodology avoids mineral acids and metal catalysts while operating under mild and solvent-free conditions. Reaction parameters such as catalyst type, temperature, and reaction time were optimized to achieve maximum yield. Among the tested bio-catalysts, Kokam juice demonstrated superior catalytic performance, affording yields up to 93% within 50 minutes at 70 °C. The protocol highlights the potential of renewable fruit-derived acids as cost-effective and biodegradable catalysts in green organic synthesis*

**Keywords:** Sustainable synthesis, Biginelli reaction, green chemistry, bio-catalyst, fruit juice catalysis, multi-component reaction, dihydropyrimidinones

## 1. Introduction

Multi-component reactions (MCRs) provide efficient synthetic routes by combining three or more reactants in a single operation. The Biginelli reaction, first reported in 1893, is a classical three-component condensation involving an aldehyde, β-ketoester, and urea/thiourea, producing 3,4-dihydropyrimidin-2(1H)-ones (DHPMs). These heterocyclic compounds possess diverse biological properties including antihypertensive, antiviral, anti-inflammatory, and anticancer activities.

Conventional Biginelli protocols frequently utilize strong mineral acids or metal-based catalysts, leading to environmental concerns and waste generation. In alignment with green chemistry principles, the search for sustainable catalytic systems has intensified. Fruit juices naturally contain organic acids such as citric acid, malic acid, ascorbic acid, and hydroxycitric acid, which can provide sufficient acidity to catalyze condensation reactions.

This study investigates fruit juices as renewable bio-catalysts for an eco-friendly Biginelli synthesis.

## 2. EXPERIMENTAL SECTION

### 2.1 Materials

Benzaldehyde (1 mmol)
Ethyl acetoacetate (1 mmol)
Urea (1.2 mmol)
Fresh fruit juices (2 mL each)
All reagents were of analytical grade and used without further purification.


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### 2.2. Methods

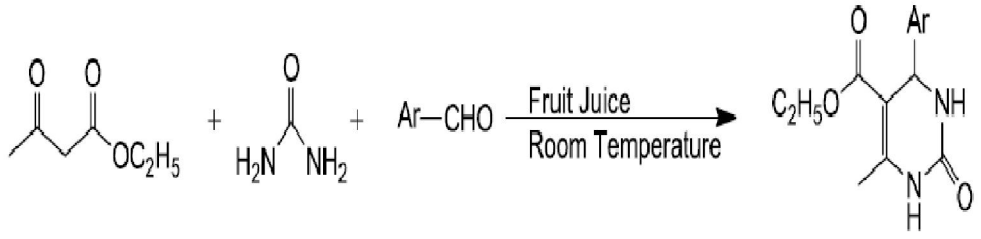
**Preparation of Fruit Juice Catalyst**

Wash the selected fruit thoroughly with distilled water.
Peel the fruit if necessary.
Extract juice using a juicer or by crushing in a mortar.
Filter the juice using muslin cloth or Whatman filter paper to remove pulp and seeds.
Use the fresh juice immediately as a bio-catalyst, or store at 4 °C for short periods (<24 h).
The pH values were measured using a digital pH meter.

**General Procedure for Biginelli Reaction Using Fruit Juice**

**Raction Setup:**

In a 50 mL round-bottom flask, add **1 mmol of aldehyde**, **1 mmol of β-ketoester**, and **1 mmol of urea/thiourea**.
Add **5–10 mL of fresh fruit juice** (volume can be optimized based on preliminary experiments).
Stir the reaction mixture at **room temperature** or slightly elevated temperature (40–60 °C) depending on the reactivity of substrates. under solvent-free conditions. Reaction progress was monitored by TLC. After completion, the mixture was cooled, poured into ice-cold water, and the precipitated product was filtered and recrystallized from ethanol.

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Mechanism

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## 3. Results and Discussion

3.1 Effect of Fruit Juice Catalyst

**Table 1. Catalytic Efficiency of Different Fruit Juices**

| Entry | Fruit Juice | pH | Time (min) | Yield (%) |
|---|---|---|---|---|
| 1 | Karwand | 3.1 | 60 | 89 |
| 2 | Kokam | 2.5 | 50 | 93 |
| 3 | Mango | 4.0 | 120 | 70 |
| 4 | Strawberry | 3.5 | 75 | 82 |
| 5 | Without catalyst | — | 180 | 28 |

Kokam juice produced the highest yield, attributed to its higher acidity and presence of hydroxycitric acid.

### 3.2 Temperature Optimization (Kokam Juice)

**Table 2. Effect of Temperature on Reaction Yield**

| Sr. No. | Temperature (°C) | Time (min) | Yield (%) |
|---|---|---|---|
| **1** | 25 | 150 | 45 |
| **2** | 50 | 90 | 78 |
| **3** | 70 | 50 | 93 |
| **4** | 80 | 50 | 94 |

The reaction at 70 °C was selected as optimal considering energy efficiency.

## 4. Characterization of Synthesized DHPM

### 4.1 Physical Properties

Product: Ethyl 4-phenyl-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate
Molecular Formula: $C_{14}H_{16}N_2O_3$
Molecular Weight: 260.29 g/mol
Melting Point: 203–205 °C
Yield: 93%

### 4.2 IR Spectral Data (KBr, $cm^{-1}$)

**Table 3. IR Spectral Analysis**

| Observed ($cm^{-1}$) | Functional Group Assignment |
|---|---|
| 3245 | N–H stretching (amide) |
| 1725 | C=O stretching (ester) |
| 1688 | C=O stretching (amide) |
| 1602 | Aromatic C=C |
| 1248 | C–N stretching |

The presence of characteristic ester and amide carbonyl absorptions confirms formation of the dihydropyrimidinone framework.

### 4.3 NMR Spectral Data

$^1$H NMR Spectral Data
(400 MHz, DMSO-$d_6$, δ ppm)

| δ (ppm) | Multiplicity | Integration | Assignment |
|---|---|---|---|
| 9.21 | s | 1H | NH (amide) |
| 7.35–7.25 | m | 5H | Aromatic protons |


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| | | | |
|---|---|---|---|
| | | | (phenyl ring) |
| 5.32 | s | 1H | C4–H (methine proton of DHPM ring) |
| 4.05 | q (J = 7.2 Hz) | 2H | $OCH_2$ (ethyl ester) |
| 2.28 | s | 3H | $CH_3$ at C6 |
| 1.15 | t (J = 7.2 Hz) | 3H | $CH_3$ of ethyl ester |

Interpretation:
The singlet at δ 9.21 ppm confirms the presence of the NH proton.
The multiplet between δ 7.35–7.25 ppm corresponds to five aromatic protons of the phenyl substituent.
The singlet at δ 5.32 ppm represents the methine proton at C4, characteristic of DHPM structures.
The quartet and triplet pattern (δ 4.05 and 1.15 ppm) confirms the ethyl ester moiety.
The singlet at δ 2.28 ppm indicates the methyl substituent at C6.
$^{13}C$ NMR Spectral Data
(100 MHz, DMSO-$d_6$, δ ppm)

| **δ (ppm)** | **Carbon Assignment** |
|---|---|
| 165.8 | Ester carbonyl (C=O) |
| 152.4 | Amide carbonyl (C=O) |
| 148.2 | C2 (pyrimidinone ring) |
| 144.6 | Aromatic quaternary carbon |
| 129.4 | Aromatic CH |
| 128.6 | Aromatic CH |
| 127.8 | Aromatic CH |
| 100.5 | C5 (olefinic carbon) |
| 60.2 | $OCH_2$ (ethyl ester) |
| 54.8 | C4 (methine carbon) |
| 18.6 | $CH_3$ at C6 |
| 14.1 | $CH_3$ of ethyl ester |

Structural Confirmation Summary
The NMR data are consistent with the proposed DHPM structure:
Two distinct carbonyl carbons observed in $^{13}C$ NMR.
Characteristic methine proton (C4–H) at ~5.3 ppm.
Ethyl ester group confirmed by quartet–triplet splitting pattern.
Aromatic signals consistent with mono-substituted phenyl ring.

**5. Proposed Mechanism**
The reaction proceeds via:
Protonation of the aldehyde by natural fruit acids.
Formation of an iminium intermediate with urea.
Nucleophilic addition of β-ketoester.
Intramolecular cyclization followed by dehydration to form DHPM.

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## 6. Green Chemistry Assessment

**Table 4. Comparison with Conventional Methods**

| Parameter | Conventional Acid Method | Fruit Juice Method |
|---|---|---|
| Catalyst | Mineral acid / metal salt | Natural organic acids |
| Solvent | Organic solvent | Solvent-free |
| Reaction Time | 2–5 hours | 50–75 min |
| Waste | Hazardous | Biodegradable |
| Toxicity | Moderate to high | Minimal |

## 7. Conclusion

An eco-friendly and efficient Biginelli synthesis has been developed using fruit juices as bio-catalysts. Kokam juice demonstrated the best catalytic activity, yielding 93% of the desired DHPM under optimized conditions. The protocol eliminates hazardous catalysts and organic solvents, aligning with green chemistry principles. This study demonstrates that naturally occurring acids in fruit extracts can serve as viable alternatives to conventional catalytic systems in multi-component organic reactions.

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