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ORIGINAL ARTICLE

Solar radiation-assisted one-pot synthesis of 3-alkylated indoles under catalyst-free conditions

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KEYWORDS

3-Alkylated Indoles;
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Abstract Solar radiation-assisted green approach was developed for the chemoselective synthesis of 3-alkylated indoles via Yonemitsu-type condensation under catalyst-free conditions. The desired products were formed by one-pot three-component reaction of indole, aromatic aldehydes, and β-dicarbonyl compounds in polyethylene glycol-400 (PEG-400). The scope and generality of the presented approach were extended by various aromatic aldehydes and C—H activated compounds. Using non-volatile solvent and renewable energy resources, absence of any catalyst, energy conservation, and the selective production of heterodimer product instead of homodimer adducts such as bisindole or xanthine, increase the economic and environmental effectiveness of the present protocol.

1. Introduction

Over the last few years, an ever-increasing concern about the environmental protection has led the chemical industry and research communities to set out alternative chemical processes that not only minimize the harmful chemicals emission but also maximize the energy conservation (Oakes, et al., 2001). Toward the decline in total required energy, some technology such as microwave, sonochemical, and UV irradiation have been utilized as a source of energy in the various chemical transformations (Chatel, 2018; Cintas and Luche, 1999). Although most of these sources offer obvious advantages, being non-renewable and non-scalable, limit the utilization of these techniques. Therefore, in recent years, special attention has been drawn to development of alternative energy sources in conjunction with non-toxic solvents, which dramatically reduce reaction time, energy consumption, and increases the chemical selectivity, or even eliminate the by-products formation.

Solar radiation is considerably appreciated due to its effect on living matter and the feasibility of its application for profitable purposes. It is the most abundant natural renewable energy resource that has great potential to accelerate chemical processes. In this respect, few chemical transformations such as oxidation of alcohol, benzylic bromi-

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nation, *N*-acylation and *N*-formylation reactions, synthesis of chalcones, benzimidazoles, triaryl pyridine derivatives, and hetero arylmethylene isoxazoles have been carried out in green and eco-friendly solvents under solar radiation (Deshpande et al., 2015; Gadilohar et al., 2016; Gadkari et al., 2020; Jadhav et al., 2017; Rathi and Shankarling, 2019; Harsh et al., 2018). Furthermore, gold, zinc oxide, and palladium nanoparticles have been successfully synthesized under solar radiation conditions (Hinge and Pandit, 2017; Nadal, et al., 2020; Patil, et al., 2012).

Nowadays, the multicomponent approach is a well-accepted strategy for the synthesis of structurally complex molecules, especially pharmacologically active organic compounds (Mojtahedi, et al., 2022; Sarmasti, et al., 2021). Indole-based compounds are one of the most valuable heterocycles, which have significant pharmaceutical and biological properties such as neuroprotective, antimicrobial, antitubercular, antimalarial, anti-tubulin, anticancer, antiviral, and antioxidant (Kardooni and Kiasat, 2018a; Ma, et al., 2021). Among the various well-known indole derivatives, 3-alkylated indoles are of significant importance due to their biological potency and crucial role in the constructing of therapeutic agents (Deb and Bhuyan, 2007; Kumar, et al., 2012; Qu, et al., 2011; Singh, et al., 2013).

The first successful report about condensation reaction of indole, aldehydes, and Meldrum's acid which afforded 3-alkylated indole reported by Yonemitsu and co-workers as about 44 years ago (Oikawa, et al., 1978). Up to now, this crucial building block has been synthesized under classical conditions such as conventional heating, (Deb and Bhuyan, 2007; Kardooni and Kiasat, 2019) microwave, (Viola, et al., 2014) or ultrasonic irradiation (Lü, et al., 2017) with the involvement of indole, aldehyde, and several β-diketones such as acetoacetates, (Viola, et al., 2014) 1,3-indandione, (Dandia, et al., 2013) nitroacetates, (Gerard, et al., 2010) 4-hydroxycoumarin, (Brahmachari and Das, 2014; Mousavizadeh, et al., 2014; Rajesh, et al., 2015) triacetic acid lactone, (Appendino, et al., 2009; Brahmachari and Das, 2014) dimedone, (Li, et al., 2014; Rajesh, et al., 2015) and N,*N*-dimethylbarbituric acid (Deb and Bhuyan, 2007) in the presence of different catalysts. Although several approaches reported for the synthesis of 3-alkylated indoles, the development of a simple, efficient, catalyst-free, and ecologically acceptable synthetic strategy remains a challenging task and in great demand.

Based on the earlier-mentioned facts and in the direction of our in progress research on the development of a green synthetic methodology for the preparation of heterocyclic compounds, (Kardooni and Kiasat, 2019, 2018b) herein we describe a catalyst-free and energy-efficient strategy for the chemoselective synthesis of 3-alkylated indoles via condensation reaction of indole, aromatic aldehydes, and activated methylene compounds in polyethylene glycol-400 (PEG-400) under concentrated solar radiation conditions.

2. Experimental

2.1. Materials and methods

The high purity reagents and solvents were purchased from Merck chemical supplier. The reaction progress monitoring was carried out with thin layered chromatography (TLC) using Merck TLC Aluminum sheets (60F254). The products were characterized by comparison of their melting point and spectral data with known samples reported in the literature. The intensity of solar radiation received at the earth's surface at Ahvaz, Khuzestan, Iran (31°19′13″, 48°40′09″) was measured for one year (2019) by a solar power meter five times on a day at 10, 11, 12, 13, and 14o'clock.

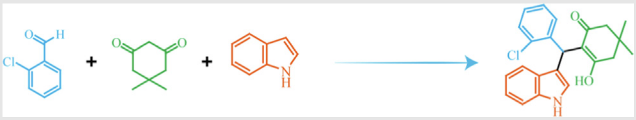
2.2. Concentrated solar radiation promoted synthesis of 3-alkylated indoles

In a typical procedure, the round bottom flask charging with indole (1 mmol), aromatic aldehyde (1 mmol), β-dicarbonyl compounds (dimedone, 4-hydroxycoumarin, or triacetic acid lactone) (1 mmol), and PEG-400 (2 ml), placed on the magnetic stirrer and fixed in the focal point of the parabolic dish for 10 min. After completion of the titled reaction, the extraction of the product was accomplished by 10 ml distilled water and (3 × 10 ml) diethyl ether. Drying the separated organic phase over anhydrous sodium sulphate, and then recrystallization of the obtained product with hot ethanol afforded the pure target product (Table 2).

3. Results and discussion

The designed solar collector system is a point-focus distributed two-axis tracking collector that includes two basic components, parabolic dish, and tower. The collector is a circular paraboloidal plate with a diameter of 80 cm covered with around 320 Hg-coated square mirrors within 4 × 4 cm^2 dimension and reflection power of 70 % which is measured with a He-Ne laser at $\lambda = 633$ nm. Additionally, three wheels and a scissor jack embed for moving and tracking the sun in both altitudes and azimuths to adjust the dish onto a reaction flask located on the tower. The tower can rotate on its base and

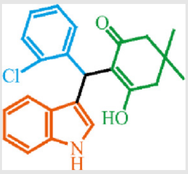
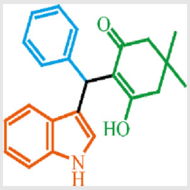
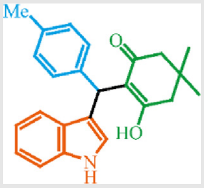
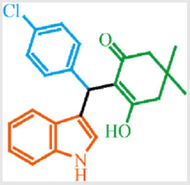
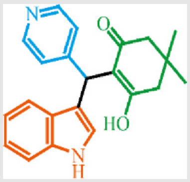
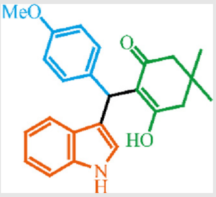
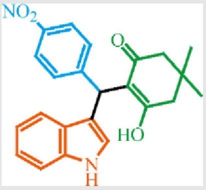
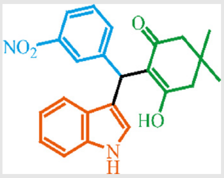
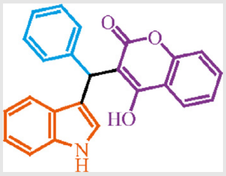
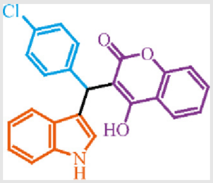
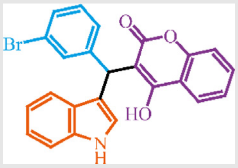
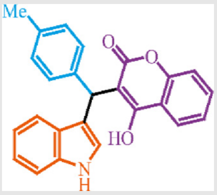
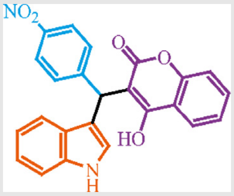
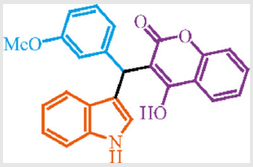
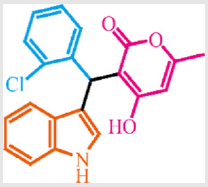
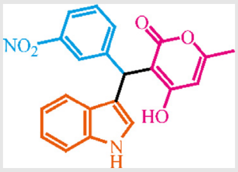
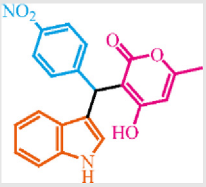
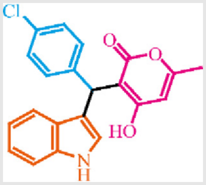
Table 1 Optimization of reaction conditions.



Entry	Reaction conditions	Result
1	Solvent-free/CSR	Decomposition of starting materials
2	Water/CSR	Incomplete reaction (90 min)
3	Water/Reflux	Contaminated with by-product (90 min)
4	PEG-400/CSR	Completed reaction (10 min)
5	PEG-400/Sunlight	Incomplete reaction (120 min)
6	PEG-400/Thermal, 70 °C	Completed reaction (60 min)

*Reaction conditions: 2- chlorobenzaldehyde (1 mmol), dimedone (1 mmol), indole (1 mmol).

Table 2 Conversion of starting materials to 3-alkylated indole.

 **$4a_1$** Time: 10 min, yield: 91 % m.p (°C): 210–212 (Rajesh, et al., 2015)	 **$4a_2$** Time: 10 min, yield: 89 % m.p (°C): 158–161 (Brahmachari and Das, 2014)	 **$4a_3$** Time: 10 min, yield: 83 % m.p (°C): 83–86 (Brahmachari and Das, 2014)	 **$4a_4$** Time: 10 min, yield: 80 % m.p (°C): 131–134 (Khan, et al., 2018)	 **$4a_5$** Time: 10 min, yield: 84 % m.p (°C): 202–203 (Li, et al., 2014)
 **$4a_6$** Time: 10 min, yield: 85 % m.p (°C): 152–155 (Khan, et al., 2018)	 **$4a_7$** Time: 10 min, yield: 90 % m.p (°C): 126–128 (Khan, et al., 2018)	 **$4a_8$** Time: 10 min, yield: 82 % m.p (°C): 137–140 (Khan, et al., 2018)	 **$4b_1$** Time: 10 min, yield: 91 % m.p (°C): 197–199 (Mousavizadeh, et al., 2014)	 **$4b_2$** Time: 10 min, yield: 82 % m.p (°C): 185–187 (Mousavizadeh, et al., 2014)
 **$4b_3$** Time: 10 min, yield: 93 % m.p (°C): 202–204 (Mousavizadeh, et al., 2014)	 **$4b_4$** Time: 10 min, yield: 88 % m.p (°C): 165–167 (Mousavizadeh, et al., 2014)	 **$4b_5$** Time: 10 min, yield: 90 % m.p (°C): 202–204 (Mousavizadeh, et al., 2014)	 **$4b_6$** Time: 10 min, yield: 84 % m.p (°C): 182 (Shaily, et al., 2016)	 **$4c_1$** Time: 10 min, yield: 85 % m.p (°C): 81–82 (Li, et al., 2014)
 **$4c_2$** Time: 10 min, yield: 90 % m.p (°C): 148–149 (-)	 **$4c_3$** Time: 10 min, yield: 85 % m.p (°C): 141–143 (Appendino, et al., 2009)	 **$4c_4$** Time: 10 min, yield: 88 % m.p (°C): 102–105 (Brahmachari and Das, 2014)		

*Reaction conditions: Aromatic aldehyde (1 mmol), β-dicarbonyl compounds (1 mmol), indole (1 mmol), PEG (2 ml) under CSR conditions.

changes the highest to adjust with the focal point (Fig. 1). Considering the optical aberrations, the disk-shaped focal point is measured in 5 cm. The intensity of concentrated solar radiation at the focal region of the collector can be calculated as:

$$I_{focal} = \frac{I_{solar} \times \eta \times A}{a}$$

Where I_{solar} assumed as the intensity of solar radiation, $A = \Pi R^2$ is the effective area of collector, $a = \Pi r^2$ is the area of the focal region and η is the reflection power. Considering geometrical specifications and optical properties of the designed reflector, the solar intensity at the focal region by I_{focal} / I_{solar} factor assessed approximately 45.

In order to investigate the effect of concentrated solar radiation on the chemoselective Yonemitsu-type condensation reaction, the synthesis reaction of 3-alkylated indoles derivatives was designed according to Scheme 1 and the reaction mixture fixed at the focal point of the parabolic dish.

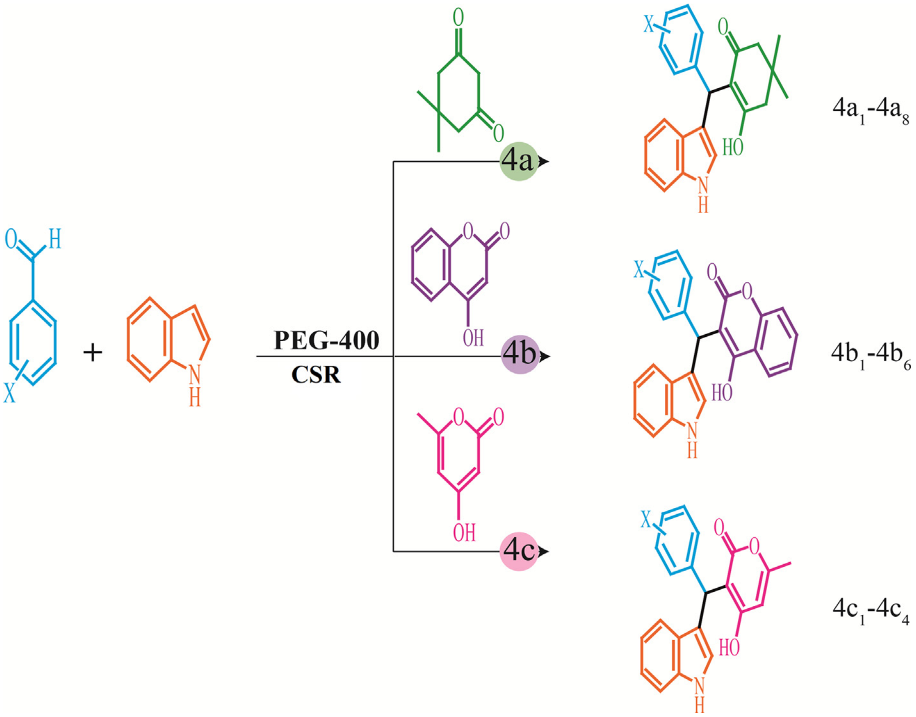
The first concentrated solar-assisted experiment was conducted with the three-components coupling reaction of indole (1.0 mmol), 2-chlorobenzaldehyde (1.0 mmol), and dimedone (1.0 mmol) under solvent-free conditions (Table 1, Entry 1). TLC analysis validates that this reaction did not lead to the formation of the desired product. This result would be justified by hot spot generation in the reaction mass and decomposition of starting materials under concentrated solar radiation (CSR).

Fig. 1 Solar collector set up.

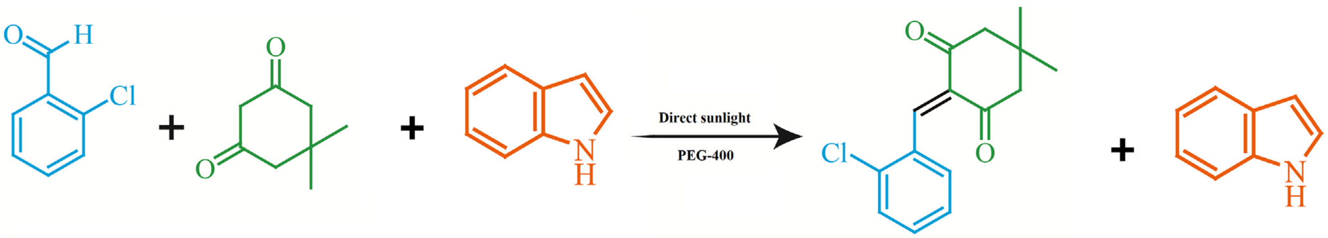
Consequently, in order to reduce the hot spot bombardment in the reaction mass and based on the green chemistry perspective, solar-assisted model reaction performed in water as a green and safe solvent (Table 1, Entry 2). Unfortunately, this reaction did not complete even after 90 min. Incompletion of this reaction would be attributed to the high thermal capacity of water which is the most significant barrier to providing the required energy for this transformation or due to the insolubility of reactants in water.

In the next experiment, to investigate whether the water is an adequate solvent for this transformation or not, the model reaction tested in water under classical reflux conditions (Table 1, Entry 3). It was observed that the desired product formed after 90 min, however, reaction media was contaminated by the generation of side products, including bisindole and xanthene. The significant reduction in reaction selectivity shows that the water is not appropriate solvent for this transformation. Considering the devastating effect of toxic and volatile organic solvents on the natural ecosystem, and green chemistry principles, PEG-400 has been chosen as a safe, non-volatile, nontoxic, biodegradable and biocompatible reaction media for performing the model reaction. Dramatically, the reaction was completed within 10 min, and the heterodimer desired product obtained in 91 % yield (Table 1, Entry 4). In the next step, to check how this transformation is affected by the concentrated solar system, the same reaction was performed in direct sunlight conditions. It was found only the Knoevenagel adduct was obtained after 120 min (Scheme 2) (Table 1, entry 5). This result underlined that the concentrated solar system has a crucial role in this conversion. As a matter of fact, the presence of Infra-red radiation in solar light caused increase in vibrational and rotational molecular motions. Thus, the bombardment of molecular species with solar radiation intensified the molecular movements, which lead to the enhancement of molecular heating. Additionally, the UV–Visible radiations which are existing in the CSR system may increase the reaction temperature due to the stimulation of starting materials. Consequently, the combined effect of IR and UV radiation provide sufficient kinetic energy for this transformation.

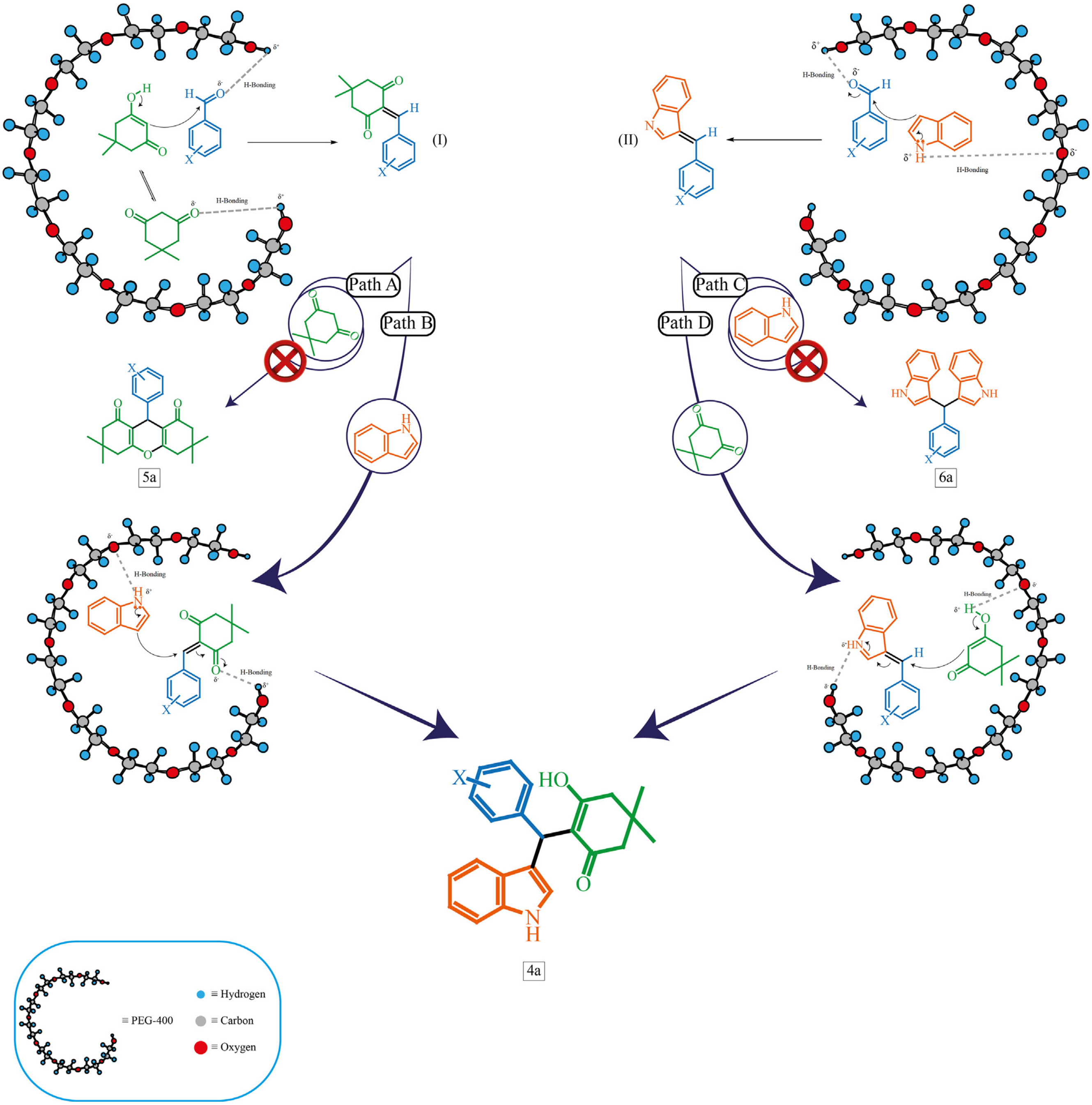
Further, to study the scope and generality of the introduced methodology in the synthesis of titled compounds, various aromatic aldehyde containing both electron-donating or electron-withdrawing substitution such as -Cl, -Me, -OMe, –NO_2, and –pyridine carbaldehyde were reacted with dimedone and indole to afford various 3-substituted indole ($4a_1$-$4a_8$) in high isolated yield and in short reaction time (Table 2). Notably, the reaction of indole and various aromatic aldehydes with other β-



Scheme 1 Chemoselective synthesis of 3-alkylated indoles under concentrated solar radiation.



Scheme 2 Knoevenagel adduct formation under direct sunlight conditions.



Scheme 3 Plausible mechanism for the synthesis of 3-substituted indoles.

dicarbonyl compounds such as 4-hydroxycoumarine and triacetic acid lactone lead to the formation of the ($4b_1$-$4b_6$) and ($4c_1$-$4c_4$) target products in a short reaction time (Table 2).

Moreover, the efficiency and practicability of this method in the synthesis of the titled compounds further demonstrated with the gram-scale reaction of indole, 2-chlorobenzaldehyde, and dimedone in PEG-400 under CSR. As expected, 2-((2-chlorophenyl) (1H-indol-3-yl) methyl)-3–hydroxy–5,5-dimethylcyclohex-2-enone was obtained in 91 % yield within 10 min.

Afterward, to find out whether the reaction mechanism is a solar photo-thermal or solar photo-radical, PEG-mediated model reaction promoted by CSR, carried out in the presence of DPPH (2,2-diphenyl-1-picrylhydrazyl) as a radical scavenger. This reaction led to the formation of the same product, 2-((2-chlorophenyl) (1H-indol-3-yl) methyl)-3–hydroxy–5,5-dimethylcyclohex-2-enone ($4a_1$), as obtained in the absence of DPPH. Therefore, it can be comprehended that the reaction proceeds in a solar photo thermal pathway.

In a plausible mechanism, the introduced reaction may proceed in four pathways (A-D). As depicted in Scheme 3, Path B and D lead to the formation of the heterodimer product, namely 3-alkylated indole. However, due to the nucleophilic activity of the indole and dimedone, the reaction may lead to the formation of homodimer products, including bisindole or xanthene in path A and path C respectively. In the course of this study, it was found that the reaction proceeds chemo selectively to afford heterodimer products.

As demonstrated in Scheme 3, the production of (4a) proceeds with the formation of Knoevenagel intermediate (I) or (П). In order to investigate the reaction pathway, and rate determinate step (RDS), PEG-mediated reaction of dimedone, 2-chlorobenzaldehyde, and indole was carried out at room temperature. It was found that only the Knoevenagel adduct (I) was obtained after 120 min. Production of the Knoevenagel adduct reveals that indole did not participate in this reaction; thus the required energy for the involvement of aldehyde and dimedone is lower than essential energy for condensation of indole and aldehyde. Therefore, it can be deduced that path B is the preferred route for the formation of (4a) and the second step, which is nucleophilic addition of indole to Knoevenagel adduct (I) is RDS in this transformation.

Moreover, it is believed that PEG served as both solvent and reaction promotor in this transformation. Considering the PEG potential to taking a helical form, it can accommodate the reactant and act as a micro vessel. In addition, PEG may form the hydrogen bonding with the starting materials by its terminal hydroxyl groups and ethereal linkage.

4. Conclusions

Briefly, solar radiation was utilized as a renewable energy source for the development of multicomponent synthesis of 3-alkylated indoles under catalyst-free conditions. This synthetic method proceeds with the involvement of indole, aromatic aldehydes, and 1,3-dicarbonyl compounds in PEG-400 as a reaction media. The outstanding features of this strategy are using a renewable source of energy, energy conservation, absence of any catalyst, using non-volatile solvent, scalability, and production of heterodimer products instead of homodimer adducts.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.arabjc.2023.104609.

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