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# Biodiesel Production from *Melia azedarach* and *Ricinus communis* Oil by Transesterification Process

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**Abstract:** Biodiesel is a renewable fuel usually produced from vegetable oils and animal fats. This study investigates the extraction of oil and its conversion into biodiesel by base-catalyzed transesterification. Firstly, the effect of various solvents (methanol, n-hexane, chloroform, di-ethyl ether) on extraction of oil from non-edible crops, such as *R. communis* and *M. azedarach*, were examined. It was observed that a higher concentration of oil was obtained from *R. communis* (43.6%) as compared to *M. azedarach* (35.6%) by using methanol and n-hexane, respectively. The extracted oils were subjected to NaOH (1%) catalyzed transesterification by analyzing the effect of oil/methanol molar ratio (1:4, 1:6, 1:8 and 1:10) and varying temperature (20, 40, 60 and 80 °C) for 2.5 h of reaction time. *M. azedarach* yielded 88% and *R. communis* yielded 93% biodiesel in 1:6 and 1:8 molar concentrations at ambient temperature whereas, 60 °C was selected as an optimum temperature, giving 90% (*M. azedarach*) and 94% (*R. communis*) biodiesel. The extracted oil and biodiesel were characterized for various parameters and most of the properties fulfilled the American Society for Testing and Materials (ASTM) standard biodiesel. The further characterization of fatty acids was done by Gas Chromatography/Mass Spectrometer (GC/MS) and oleic acid was found to be dominant in *M. azedarach* (61.5%) and *R. communis* contained ricinoleic acid (75.53%). Furthermore, the functional groups were analyzed by Fourier Transform Infrared Spectroscopy. The results suggested that both of the oils are easily available and can be used for commercial biodiesel production at a cost-effective scale.

**Keywords:** *Melia azedarach*; *Ricinus communis*; transesterification; fatty acid; biodiesel

## 1. Introduction

Impeding fossil fuels are of major concern due to their increasing consumption every day. Utilization of these fuels is also bringing less supply of fossil energy and destruction of many environments [1,2]. The CO<sub>2</sub> emission due to fossil fuels is a major cause of global warming. Various chemical, physical and biological methods have been implemented to capture CO<sub>2</sub> [3]. Because of increasing energy demand, alternative fuel, such as biodiesel, shows a great potential to fulfill this

need of energy [4]. Biodiesel was announced as a good substitute for traditional diesel because it is biodegradable, non-toxic, eco-friendly and renewable. Biodiesel has lower Sulphur content (0.0018 wt. %) that is 28-fold less than diesel fuel (0.0500 wt. %) [5]. Chemically, biodiesel is composed of alky methyl esters of long chain fatty acids produced through fatty acid esterification or transesterification of cooking oils, animal fats and vegetable oils [6,7].

Through the transesterification process, vegetable oils are converted into esters in the presence of an alcohol. Glycerol is produced as a byproduct during this reaction and it has a contribution in cosmetic products and pharmaceutical sciences [8]. This reaction performs better in the presence of homogeneous catalysts, such as sodium hydroxide (NaOH) or sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and heterogeneous catalysts like carbonates or metal oxides. Moreover, sodium hydroxide is widely used because it is cheap and yields high amounts of product. Among the alcohol used, methanol is frequently used because of its low cost [9]. The reaction catalyzed by an alkali is much faster and used at commercial level [10].

The biodiesel can be produced from various non-edible oils extracted from plants available in the world and can serve as a potential energy source for future energy demands. Non-edible crops include *Heveabrasinensis* (rubber tree), *Jatropha curcas* (ratanjot), *S. chinensis* (jojoba), *Melia azedarach* (syringa), *Ricinus communis* (castor), *Calophylluminophyllum* (polanga) and *Pongamiapinnata* (karanj). These crops can be grown in an area which is generally not suitable for cultivating food crops and these are more environmentally friendly, efficient and produce more economic products as compared to edible oils [11]. Among non-edible sources, the seeds of jatropha, castor and microalgal oil are considered as the most reliable sources which have high oil content [12]. Algae are cheap sources of biodiesel due to low cost and easy availability [13]. The berries of *Melia azedarach* contain about 10% oil and can be extracted by organic solvent and converted into biodiesel. The oil of *M. azedarach* contains fatty acids such as oleic (21.8 wt. %) acid, linoleic (64.1 wt. %) acid, stearic (3.5 wt %) acids and palmitic (10.1 wt. %) acids [14]. The extracted castor oil contains 50% oil and it contains a high amount of fatty acids. The high content of ricinoleic acid (85%) makes it different from other oils on the basis of high polarity exhibited by ricinoleic acid; it is the only oil soluble in alcohol at room temperature. The castor bean plant has attained an attention because of its less production cost and easy growth. Its seeds contain 53% oil contents and has a productivity of 1180 kg oil per hectare [15]. Therefore, the present study focused on the process of oil extraction from *Melia azedarach* and *Ricinus communis* seeds by using various solvents; the effects of various variables on biodiesel yield were optimized during the transesterification process. Moreover, GC and FTIR analysis was used for characterization.

## 2. Results and Discussion

### 2.1. Biochemical Composition of Samples

The composition of biomass samples (*M. azedarach* and *R. communis*) was analyzed and shown in Table 1. The *M. azedarach* fruit exhibited higher moisture content (5.8%) as compared to the seeds of *R. communis* (3.1%). Moreover, higher fat contents were recorded in *R. communis* (60.5 %) than *M. azedarach* (51.9%) and crude fiber (12.2%) and carbohydrate (8.5%) contents were comparatively higher in *M. azedarach*. These findings agree with a previous study that reported that *R. communis* had more protein and fat contents than other related species [16]. Similarly, in one of the study, significantly higher fat contents (40.2%), crude fibre (22.0%) and protein (20.8%) were recorded in *R. communis* [17].

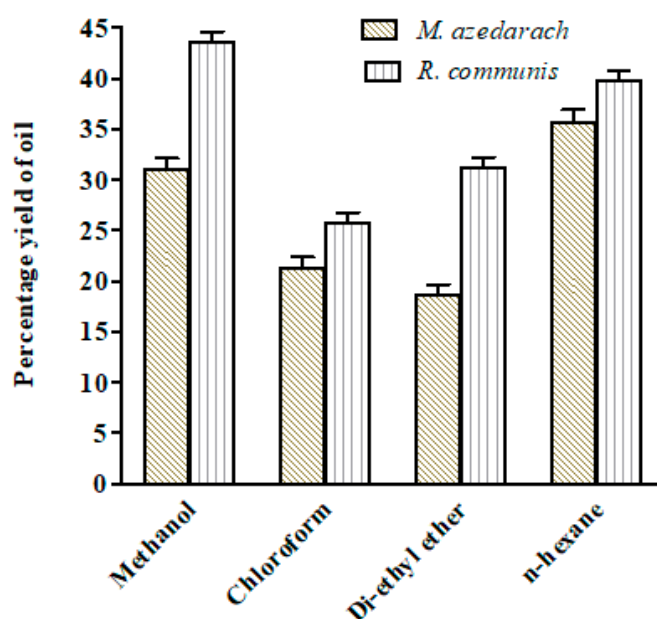
### 2.2. Oil Extraction from Biomass Samples by Solvent Extraction Method

Oil contents of *M. azedarach* and *R. communis* extracted by using different solvents (methanol, n-hexane, and chloroform and di-ethyl ether) are shown in Figure 1. The oil contents of *M. azedarach* obtained from soxhlet apparatus varied between 18.7% (di-ethyl ether) and 35.6% (n-hexane). On the other hand, oil contents of *R. communis* seeds were changed between 25.80% (chloroform) and 43.6% (methanol). Regarding the effect of solvents on oil extraction, the maximum yield was obtained with

methanol (*R. communis*, 43.6%) and n-hexane (*M. azedarach*, 35.6%). Hence, it was concluded that the polarity of solvents has influence on extraction of oil from biomass.

**Table 1.** Compositional analysis of biodiesel feedstocks.

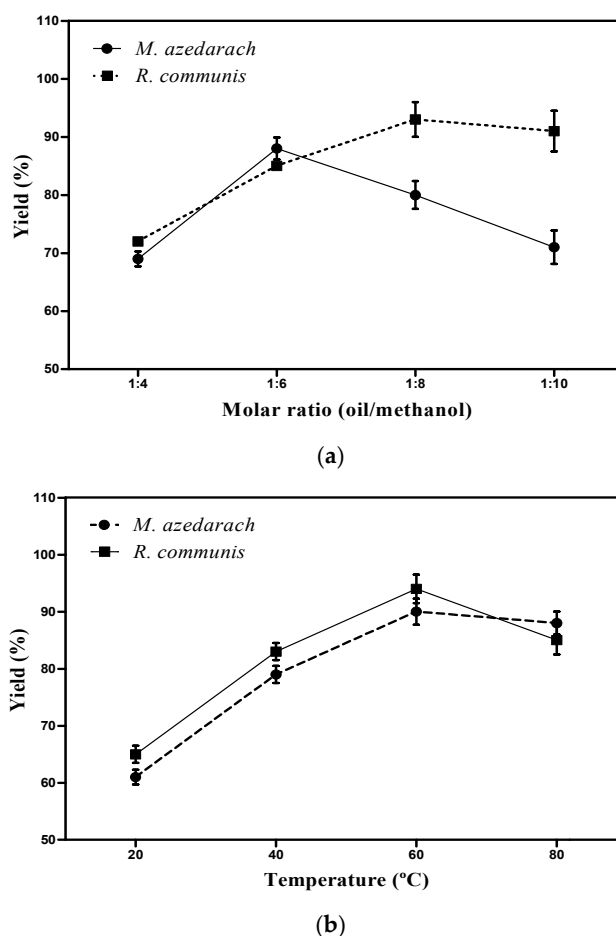
Parameters	<i>Melia azedarach</i>	<i>Ricinus communis</i>
Dry matter (%)	94.2 ± 0.1	96.9 ± 0.0
Moisture (%)	5.8 ± 0.0	3.1 ± 0.1
Crude protein (%)	14.0 ± 0.3	15.3 ± 0.2
Crude fat (%)	51.9 ± 0.2	60.5 ± 0.5
Crude fiber (%)	12.2 ± 0.1	9.6 ± 0.1
Carbohydrate (%)	8.5 ± 0.1	5.8 ± 0.5
Ash (%)	5.4 ± 0.5	2.9 ± 0.4



**Figure 1.** Comparison of oil extracted from biomass samples by using different solvents (Organized by Decreasing Polarity Index).

### 2.3. Base Catalyzed Transesterification of Extracted Oil into Biodiesel

In order to check the effect of alcohol during transesterification on biodiesel yield, various oil/methanol molar ratios (1:4, 1:6, 1:8 and 1:10) were selected. Other reaction parameters were kept constant (temperature, 60 °C; reaction time, 2.5 h; and concentration of catalyst, 1% NaOH). Figure 2a shows the change in ester contents as a function of the oil/methanol molar ratio. A ratio of 1:6 was selected in case of *M. azedarach* as it yield 88% fatty acid methyl ester (FAME) contents, whereas the higher biodiesel yield (93%) was achieved at a ratio of 1:8 in *R. communis*. At a high concentration of methanol, the biodiesel yield was decreased. Moreover, at higher methanol concentration the products has a difficulty of separation and longer time will be required [18]. Figure 2b gives the ester contents of *M. azedarach* and *R. communis* at four different reaction temperatures (20, 40, 60 and 80 °C) by keeping the other variables constant and the highest yield was obtained at 60 °C in both of the samples (90% and 94%, respectively). In a previous study, the highest yield of biodiesel was obtained at 65 °C because of the acceleration of the chemical reaction due to increased collision of the reactant molecules and miscibility [19].



**Figure 2.** Conversion of oil into biodiesel (a) Effect of oil/methanol molar ratio on biodiesel yield (b) Effect of varying temperature on biodiesel yield.

## 2.4. Characterization of Oil and Biodiesel

### 2.4.1. Physico-Chemical Characteristics

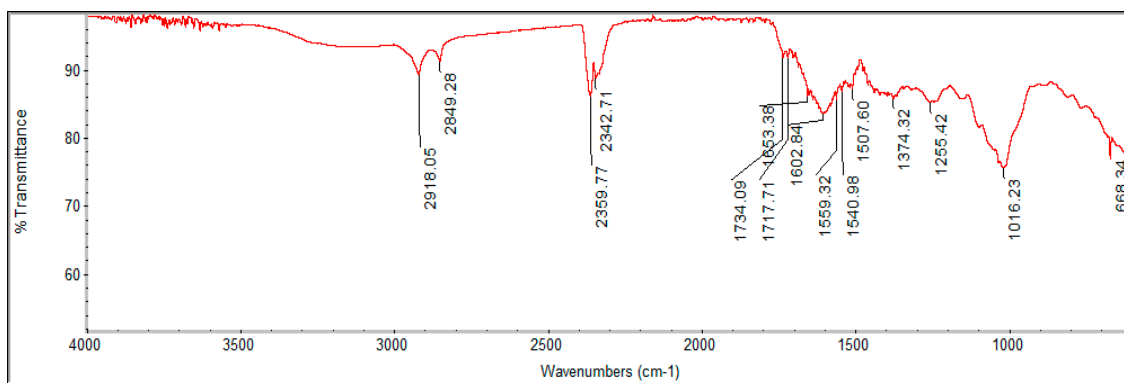
Table 2 Shows the properties of oil as well as biodiesel in *M. azedarach* and *R. communis* samples at optimized conditions. Density affects the engine performance and the density of biodiesel depends on ester contents and remaining alcohol quantity [20]. The oils of selected biomasses have higher kinematic viscosity and the higher kinematic viscosity of *R. communis* is caused by the presence of hydroxyl groups [21]; after transesterification a higher reduction of viscosity was recorded. As mentioned in the table, most of the biodiesel properties meet the requirements of standard ASTM (American society of testing material) biodiesel.

### 2.4.2. FTIR Analysis and Fatty Acid Profiling by GC

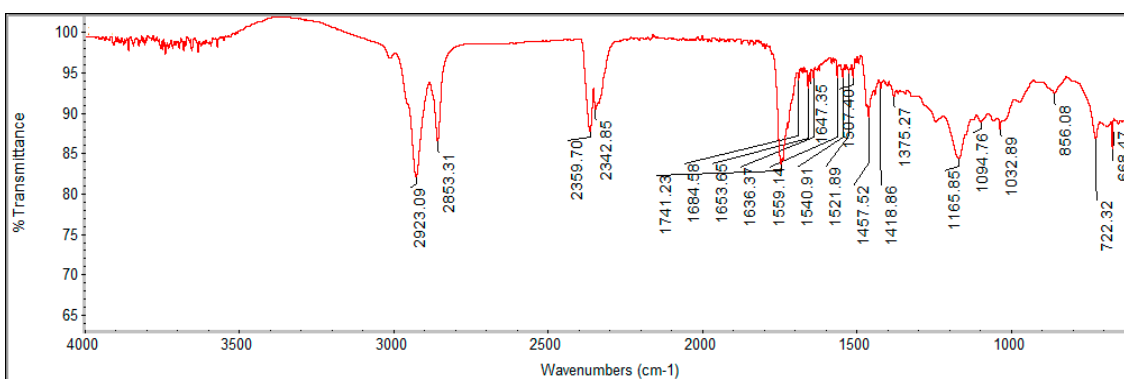
FT-IR is a precise and rapid method for the determination of functional groups in extracted oil as well as in biodiesel [22]. The most characteristic absorption peaks of *R. communis* and *M. azedarach* oil are represented in Figure 3a,b, respectively. The absorption peaks appearing at  $2923.09\text{ cm}^{-1}$  and  $2853.31\text{ cm}^{-1}$  indicate  $\text{CH}_2$  stretching and the other sharp peaks at  $1165.85\text{ cm}^{-1}$  and  $1741.23\text{ cm}^{-1}$  represent  $\text{C}=\text{O}$  aldehyde and ester stretch in Figure 3a. Moreover, Figure 3b shows the absorption peaks of the produced biodiesel of *M. azedarach* at  $1076.53\text{ cm}^{-1}$  and  $1734\text{ cm}^{-1}$  indicates  $\text{C}=\text{O}$  aldehyde and ester groups. The characterization peak at  $1374.32\text{ cm}^{-1}$  corresponds to  $-\text{CH}_3$  (Alkyl) bending.

**Table 2.** Physico-chemical characteristics of oil and fuel properties of biodiesel.

Parameters	<i>Melia azedarach</i>		<i>Ricinus communis</i>	
	Oil	Biodiesel	Oil	Biodiesel
Acid value (mg KOH/g oil)	5.4 ± 0.1	0.9 ± 0.0	2.0 ± 0.0	0.8 ± 0.0
Saponification value (mg KOH/g oil)	171.8 ± 0.2	48.9 ± 0.5	174.9 ± 0.7	181.3 ± 0.4
Iodine value (g I <sub>2</sub> 100 g <sup>-1</sup> )	127.2 ± 0.3	119.0 ± 0.2	83.5 ± 0.2	81.3 ± 0.3
Cetane number	-	49.2 ± 0.5	-	41.2 ± 0.1
Density <sub>15oC</sub> (g/cm <sup>3</sup> )	0.9 ± 0.0	0.9 ± 0.0	0.9 ± 0.0	0.9 ± 0.0
Specific gravity (g/mL)	0.95	0.89	0.94	0.87
Kinematic viscosity (mm <sup>2</sup> /s)	18.1 ± 0.1	3.2 ± 0.0	22.1 ± 0.1	11.1 ± 0.1
Refractive index (30 °C)	1.4 ± 0.0	1.4 ± 0.0	1.5 ± 0.0	1.5 ± 0.0
Cloud point	-	< -10	-	< -10
Pour point	-	-28	-	-31
Flash point	-	131.3 ± 0.5	-	261.2 ± 1.3
Calorific value (MJ/kg)	-	33.1 ± 0.4	-	37.9 ± 1.8
Cold filter plugging point	-	-30	-	-35
Sulfur content (%)	-	<0.001	-	<0.001
Free fatty acid	0.6 ± 0.0	0.3 ± 0.0	0.7 ± 0.0	0.3 ± 0.0



(a)



(b)

**Figure 3.** (a) FTIR spectrum of extracted Biodiesel of *R. communis* (b) FTIR spectrum of extracted Biodiesel of *M. azedarach*.

The extracted oils from the samples were analyzed by gas chromatography (GC). The fatty acid composition of *M. azedarach* and *R. communis* is presented in Table 3. Oleic acid contents of *M. azedarach* show  $61.5 \pm 0.4\%$  of whole oil, whereas *R. communis* has high ricinoleic acid content ( $72.53 \pm 0.7\%$ ). The fatty acid composition of biodiesel is an important parameter as it shows the efficiency of the process. It was reported that the type as well as fatty acid composition mainly depends on the selected species of plant and its cultivated condition [23]. The straight chain fatty acids are main components

of biodiesel and the most commonly used fatty acids are stearic acid (C18:0), linoleic acid (C18:2), linolenic acid (C18:3), palmitic acid (C16:0) and oleic acid (C18:1) [24]. It was previously observed that, because of high contents of monounsaturated fatty acids in biodiesel, it has better properties of fuel stability, ignition quality and flow properties at low temperatures [25,26]. Moreover, the fatty acid profiling of different non-edible oils was found to be appropriate for biodiesel production [23].

**Table 3.** Major fatty acids of extracted oils by Gas Chromatography.

Oil	Palmitic acid (C16:0)	Palmitoleic acid (C16:1)	Stearic acid (C18:0)	Oleic acid (C18:1)	Ricinoleic acid (C18:1 OH)	Linoleic acid (C18:2)	Linolenic acid (C18:3)
<i>Melia azedarach</i>	9.1 ± 0.5	1.8 ± 0.01	3.9 ± 0.1	61.5 ± 0.4	-	9.2 ± 0.2	0.8 ± 0.0
<i>Ricinus communis</i>	1.5 ± 0.1	0.9 ± 0.0	1.8 ± 0.1	6.0 ± 0.0	72.5 ± 0.7	5.1 ± 0.0	1.7 ± 0.1

### 3. Materials and Methods

#### 3.1. Collection and Preparation of Samples

*Ricinus communis* and the fruit of *Melia azedarach* were harvested from different sites of district Rawalpindi and the federal capital Islamabad. All the collected samples were collected and stored in labeled sealed plastic bags with identifications by professional taxonomists. The seeds of castor beans were separated from nibs and the fruit sample of *M. azedarach* was washed for cleaning and removal of dust and chemical materials. Drying of samples was carried out in the shade followed by oven drying at 60 °C for 7 h to remove moisture contents. An electrically powered mill was used to crush samples into fine powder and preservation was done in fine plastic bags at 4 °C till further use.

#### 3.2. Extraction of Oil by Soxhlet Apparatus

The sample of *M. azedarach* (15 g) and *R. communis* (5 g) was weighed and placed in a clean dry extraction thimble and fixed it under the condenser. Various organic solvents like chloroform, methanol, and n-hexane and di-ethyl ether were used for oil extraction. A total of 300 mL of each solvent were taken into round bottom flasks and connected with a continuous supply of water for condensation. At a constant temperature (60 °C), the flask was continuously heated to reflux. After boiling, the solvent was continuously evaporated from thimble into the condenser and the vapors were sent in the thimble. The extract was passed through the pores of thimble filter paper, filled the siphon tube and flowed into the receiving flask. This process took 30 min to complete and was repeated five to six times. Moreover, the used solvent was recovered by rotary evaporator to purify the oil and extracted product was oven dried and weighed to determine the oil contents. The experiment with each solvent was carried out thrice and the resultant oil was kept in a close container for further analysis [27].

#### 3.3. Base Catalyzed Transesterification

The standard procedure with modification [28] was employed, and refined oil was converted into fatty acid methyl esters. Later on, methanol with 1% NaOH solution was taken in a batch reactor (250 mL) that was attached with a reflux condenser. The oil was subsequently added for 2.5 h and reaction parameters such as temperature (20, 40, 50 and 60 °C) and methanol/oil ratio (1:2, 1:4, 1:6 and 1:8) were adjusted. Moreover, the reaction mixture was subjected to pass through a separating funnel for separation phases. All the impurities and methyl esters were trapped in the upper phase, while the lower phase contained rough glycerin etc. Before further analysis, centrifugation of biodiesel was done for 10 min at 4000 rpm.



### 3.4. Analytical Methods

The characteristics of oil and fuel properties of biodiesel were tested according to ASTM standards (ASTM, 2000).

### 3.5. Fatty Acid Analysis by GC/MS

GC/MS of the extracted oils was carried out for fatty acid profiling using a Shimadzu QP2010 gas chromatography coupled with mass spectroscopy detector (Shimadzu, Kyoto, Japan). A temperature range of 70 °C to 280 °C was set at and a carrier gas Helium was used. The injection volume was set to be 2 µL at 250 °C with a flow rate of 1.80 mL/min. ACQ scanner having 30–700 amu range at speed of 1478 was operated for mass spectroscopy (Shimadzu, Kyoto, Japan). NIST05 mass spectral library (NIST, 2012) was used as a standard for comparison of spectral data obtained after the analysis [29].

### 3.6. FTIR Analysis of Biodiesel

A total of 25 scans for each sample were conducted by ATR-FTIR background-corrected spectra with an HATR trough; a cut of 45° presented approximately ten internal reflections. Moreover, the crystal plates were kept at 20 °C, and 670-IR spectrometer was used for recording of data (Agilent Technologies, Santa Clara, CA, USA). The ultra-pure organic solvents (Sigma-Aldrich, Darmstadt, Germany) were employed to wash Ge crystal. The continuous purging of argon was done for 40 min before and also while doing measurements. Scans were Fourier-transformed and averaged with GRAMS/AI 8.0 software (Thermo Electron Corporation; Waltham, MA, United States, USA, 2014) [30].

## 4. Conclusions

This study focused on the comparative analysis of biodiesel production from non-edible oil crops, such as *R. communis* and *M. azedarach*. Organic solvents were used for oil extraction and in the presence of methanol; the yield of biodiesel was higher in *R. communis* (43.60%) than *M. azedarach* (35.60%) by using n-hexane. Moreover, the conditions were optimized for base-catalyzed transesterification and at a respective concentration of 1% NaOH, 60 °C temperature, 2.5 h reaction time and 1:8 molar oil/methanol molar ratio, *R. communis* yields highest biodiesel yield (94%) as compared to *M. azedarach* (90%) in the presence of 1:6 oil/methanol molar ratio. The produced biodiesel meets the requirements of international standard ASTM biodiesel. The analysis of results by GC/MS and FTIR strongly recommend that these crops are promising feedstocks for the production of biodiesel.

**Author Contributions:** Conceptualization and methodology of this research was designed and discussed by M.A., I.T. and S.A.M. Moreover, write up and revisions were conducted by M.A. and S.A.M. Software based analysis and validation of formal analysis, was done by F.J., S.U.K. and M.A.R. While the experiments were conducted by F.K. and I.B. All authors have read and agreed to the published version of the manuscript.

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