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ROUTE OF BIOFUEL PRODUCTION FROM MACADAMIA NUT SHELLS: EFFECT OF PARAMETERS ON THE PARTICLES MIXING INDEX IN FLUIDIZED BEDS

ROTA DE PRODUÇÃO DE BIOCOMBUSTÍVEL A PARTIR DE CASCA DE MACADÂMIA: EFEITO DE PARÂMETROS SOBRE O ÍNDICE DE MISTURA EM LEITOS FLUIDIZADOS

RUTA DE PRODUCCIÓN DE BIOCOMBUSTIBLES A PARTIR DE CORTEZA DE MACADAMIA: EFECTO DE LOS PARÁMETROS EN EL ÍNDICE DE MEZCLA DEL LECHOS FLUIDIZADOS

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ABSTRACT

Pyrolysis of macadamia nut shells (MNS) in a fluidized bed reactor has excellent potential to produce bio-oil. High heat transfer rates and uniform temperature in the fluidized bed can be achieved due to effective gas-solid contact in the reactor. However, binary mixtures can lead to the segregation of particles, which negatively affects heat and mass transfer in such a reactor. Therefore, a 2^3 statistical experimental design was used to assess the effects of parameters (i.e., air velocity, particle diameter ratio, and mass fraction of MNS) on the mixing index of the bed of MNS and sand. Among the analyzed factors, only D_{MNS}/D_S and V/V_{MF} influenced the mixing index (I_m) within a confidence interval of 95%. Based on statistical data analysis, an air velocity 20% above the minimum fluidization and particle diameter ratio (D_{MNS}/D_S) smaller than 3 results in uniform particle mixing in the bed (i.e., reaching ideal mixing index values). Moreover, the experimental results indicate that fluidized be used for biofuel production from Macadamia nut Shells.

RESUMO

A pirólise da casca da macadâmia (MNS) em leito fluidizado tem grande potencial para produção de bio-óleo. O efetivo contato gás-sólido permite alcançar altas taxas de transferência de calor e temperatura uniforme no leito. Entretanto, misturas binárias podem levar à segregação de partículas, reduzindo as taxas de transferência de calor e massa no reator. Portanto, nesta pesquisa, o planejamento de experimentos foi aplicado para avaliar o efeito dos parâmetros (velocidade de injeção do ar, V/V_{MF}; razão do diâmetro das partículas, D_{MNS}/D_S, e fração mássica de MNS, X_{MNS}) sobre o índice de mistura (I_m) em leito fluidizado composto por MNS e areia. A análise estatística revelou que apenas D_{MNS}/D_S e V/V_{MF} tiveram efeito sobre índice de mistura (I_m), considerando um intervalo de confiança de 95%. Desta forma, para velocidade do ar 20% acima da mínima fluidização e misturas com $D_{MNS}/D_S < 3$, os leitos fluidizados apresentaram concentrações de partículas uniforme ao longo da coluna. Os resultados indicam que o leito fluidizado pode ser empregado na produção de biocombustível a partir da casca de macadâmia.

RESUMEN

La pirolisis de corteza de macadâmia (MNS) en un lecho fluidizado es un potencial para producir bio-aceite. El contacto efectivo gas-solido alcanza altas tasas de transferencia de calor y temperatura uniforme en el lecho. Aunque, las mezclas binarias pueden segregar partículas, eso reduce las tasas de transferencia de calor y masa. Por eso, se aplicó el diseño experimental para evaluar el efecto de los parámetros (velocidad de inyección del aire, V/VMF; relación del diámetro de partículas, DMNS/DS y fracción de masa, XMNS) en el índice de mezcla (Im) del lecho fluidizado compuesto por MNS y arena. El análisis de los datos reveló que solo DMNS/DS y V/VMF influenciaron en el índice de mezcla (Im), considerando un intervalo de confianza del 95%. Así, para velocidades de aire 20% arriba de la fluidización mínima y mezclas con DMNS/DS < 3, los lechos fluidizados mostraron concentraciones de partículas uniformes en toda la columna. Los resultados indican que el lecho fluidizado puede utilizarse en la producción de biocombustible a partir de la corteza de macadâmia.

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1. INTRODUCTION

Due to the increasing energy demand, depletion of fossil fuels, and high emission of greenhouse gases, more attention has been paid to the search for alternative energy sources (Massaro Sousa & Ferreira, 2020; Soria-Verdugo *et al*., 2020, 2023b; Yang *et al*., 2022; Zhou *et al*., 2022).

Among the energy sources, biomass is one of the most important alternatives due to its renewability, availability, and low cost (Lee *et al*., 2023). In this scenario, macadamia nut shells (MNS) are a promising alternative for bio-oil production (Hasan *et al*., 2022).

Macadamia nut shells are a by-product of macadamia nut processing, with 42,150 tons of throughput annually (Gong & Pegg, 2015). Each ton of macadamia nut produces about 70 to 77% shell residue (Xavier *et al*., 2016) and has more than 80% volatile material and less than 1% ash (Strezov *et al*., 2007). This biomass's use for energy production is justified by its high heating value compared to other biomasses. The heating value of MNS is about 20.01 MJ/kg, while sugarcane has 17.41 MJ/kg (Parikh *et al*., 2005). However, this biomass is commonly used as a natural fertilizer or energy source or discarded (Samoraj *et al*., 2022; Yang *et al*., 2022).

Physicochemical, biochemical, and thermo-chemical processes can convert biomass waste. Thermochemical conversion of biomass wastes includes combustion, gasification, pyrolysis, hydrothermal liquefaction, and hydrothermal carbonization (Soria-Verdugo et al., 2023a). Among the thermochemical processes for biomass waste conversion, pyrolysis is a promising and advantageous route to obtain high-added-value products (Mantegazini *et al*., 2021; Tchoffor *et al*., 2015; Tran *et al*., 2020; Zhou *et al*., 2022).

Various reaction systems (fixed bed, fluidized bed, and conical spouted bed) have been investigated for biomass pyrolysis. For large-scale bio-oil production, fluidized bed reactors have been highlighted by achieving uniform bed temperature due to intense gas-solid contact and good particle mixing (Ji *et al*., 2016; Martinez Castilla *et al*., 2020; Wang *et al*., 2022; Wang & Shen, 2021). Due to the low density of biomass and poor flowability, sand is added as an inert to improve the fluid dynamic stability and heat transfer of either the fluidized bed or conical spouted bed reactors (Artetxe *et al*., 2010; Barcelos *et al*., 2020; Freitas *et al*., 2017; López *et al*., 2010; Marques & Bacelos, 2013; Melo *et al*., 2016)

As noted in the Literature, binary mixtures can lead to particle segregation due to different sizes and densities (Bacelos & Freire, 2006; Barcelos *et al*., 2020; Daleffe & Freire, 2004; Hidaka *et al*., 1995; José *et al*., 1994a; Norouzi *et al*., 2012; Saidi *et al*., 2014; Selvatici *et al*., 2021).

Therefore, segregation affects particle distribution, heat, and mass transfer rates in bed, consequently leading to hot spots in fluidized bed reactors. However, this can be avoided by adjusting process parameters (such as fluidization velocity), bed composition, and particle size. For contributing to biofuel production routes from biomass wastes, this research paper aims to assess the effects of air velocity, particle diameter ratio, and mass fraction of MNS on the bed mixing index achieved in a fluidized bed of macadamia nut shells and sand mixtures.

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Moreover, this research not only intends to point out the limits of air velocity but also shows parameter limits of mixture composition and particle size, achieving uniform particle concentration in the bed column. Such parameter limits are essential, especially for reactor start-ups in pyrolysis plants for biofuel production (Iannello *et al*., 2023; Lopez *et al*., 2017).

2. MATERIALS AND METHODS

2.1. MATERIAL

For particle segregation analysis in the fluidized bed reactor, mixtures of macadamia nut shells (MNS) and sand were selected, as shown in Figure 1.

Figure 1. Particles: (a) macadamia nut shells (MNS), and (b) sand.

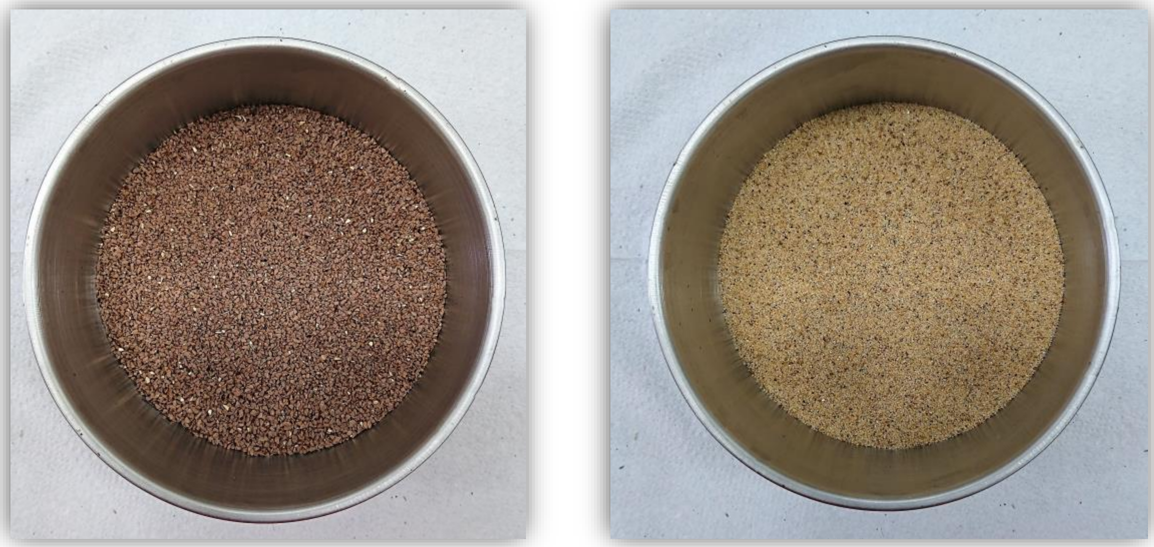

Table 1 shows the macadamia nut shells (MNS) and sand particle size and apparent density) obtained by sieving and liquid picnometry, respectively. Kerosene was used to determine MNS apparent density, and water was employed to obtain sand particles' apparent density.

Table 1. Properties of macadamia nut shells (MNS) and sand particles.

Material	Apparent density (ρ) (kg/m³)	Particle diameter (mm)
MNS	1180	0.7 1.3
Sand	2510	0.4

2.2. EXPERIMENTAL APPARATUS

A cylindrical fluidized bed column of MSN and sand particles was used to study the particle concentration. Figure 2 shows a schematic of the experimental apparatus found in the Energy Efficiency Laboratory from the Postgraduate Program in Energy (PPGEN) of CEUNES/UFES.

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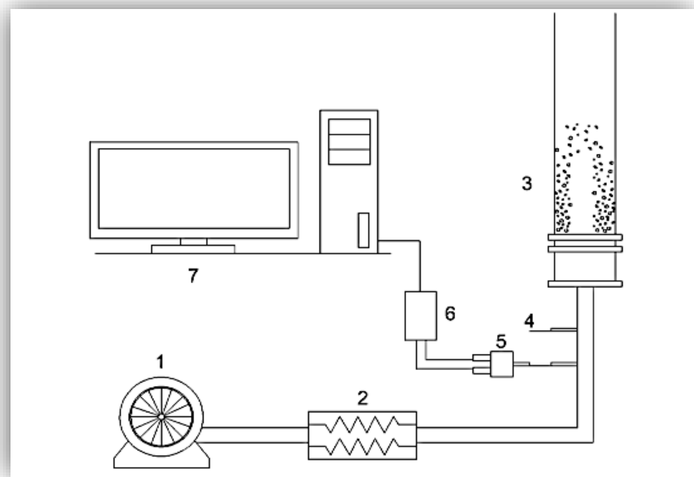
The fluidized bed system (Figure 2) consisted of a 2.0 hp centrifugal blower with a 4.5 m^3/min maximum air flow rate by IBRAM, an air heating system to control an air inlet temperature, a pressure transducer by Dwyer to measure inlet pressure (Dwyer 616C-4).

An A/D data acquisition card (CDAQ-9174) by National Instruments acquired the sensor's analog signals. Then, LabVIEW 7 software to a computer (Core i3, 3.30GHz, 4GB RAM) processes the sensor signals connected. A thermal anemometer probe (AMI 300) by Kimo Instruments measured the superficial air velocity of the column at the top of the column.

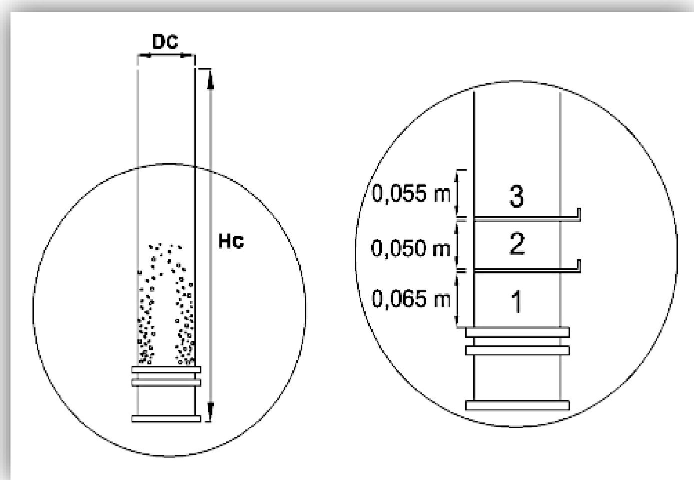
Figure 2. Fluidized bed unit: 1 - blower; 2 - air heating system; 3 - column; 4 - thermocouple; 5 - pressure transducer; 6 - data acquisition board; 7 - computer.



Source: (Selvatici *et al*., 2021).

The column used has the following geometrical parameters: 0.1 m column diameter (Dc), and 0.72 m column height (Hc). The column was equipped with two guillotines by Selvatici and co-workers (Selvatici *et al*., 2021) to evaluate particle segregation. The guillotines allow the collection of particle mixture samples at different axial positions, as shown in Figure 3.

Figure 3. Column dimensions and guillotines position.



Source: (Selvatici *et al*., 2021).

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2.3. EXPERIMENTAL PROCEDURE

2.3.1. Design of experiments

A 2^3 experimental design was used to analyze the effects of factors (i.e., air velocity, particle diameter ratio, and mass fraction of MNS) on the mixing index. The factor values were selected based on the particles' properties and the reactor's operating conditions. A %5 significance level was chosen as the average experimental deviation ranged from 0.04 to 0.1. As expected, Region 1, located at the bed bottom, close to the air entrance, had a higher observed deviation. R1 was greatly perturbed by air flowing through the bed. Table 2 shows the experimental design. The D_{MNS}/D_S represents the diameter ratio between the macadamia shell and sand particles; X_{MNS} refers to the perceptual mass fraction of the macadamia shell, and V/V_{MF} is the ratio of a given air velocity added into the bed and the minimum fluidization velocity.

Table 2. Experimental design.

Experiments	D_{MNS}/D_S	X_{MNS} (%W/W)	V/V_{MF}
01	2.0	5.0	1.1
02	2.0	5.0	1.2
03	2.0	15.0	1.1
04	2.0	15.0	1.2
05	3.0	5.0	1.1
06	3.0	5.0	1.2
07	3.0	15.0	1.1
08	3.0	15.0	1.2

2.3.2. Fluidization

For the experimental conditions shown in Table 2, the minimum fluidization condition was determined by analyzing the characteristic curve (a plot of pressure drop versus decreasing air velocity). The minimum fluidization condition was evident from the transparent column wall.

Initially, the bed was filled with a given mixture of particles according to the experimental design. Then, the air velocity in the bed was increased until reaching a value of 10% higher than the minimum fluidization. After the bed was kept under operating conditions for 5 minutes, the air velocity was reduced. Preliminary tests showed that a five-time interval was enough to ensure the bed could reach the dynamic stationary-fluidization regime. Therefore, particles could achieve the same average concentration in the bed for a given column region.

2.3.3. Response variable

As shown in Figure 3, two guillotines were used to divide the bed into three distinct axial regions. The guillotines were inserted into the column one after the other, dividing the bed into three regions: top (region 1- R1), middle (region 2- R2), and bottom (region 3- R3). The mixtures for each region were separated by sieving. The MNS mass fraction was determined by equation 1.

$$X_{MNS,Ri} = \frac{M_{MNS,Ri}}{M_{MNS,Ri} + M_{S,Ri}} \quad (1)$$

Where, $M_{MNS,Ri}$ is the macadamia nut shells (MNS) mass for a given bed region, $M_{S,Ri}$ is the sand mass for a given bed region, and i refers to a given bed region.

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For particle segregation in fluidized bed reactors, the mixing index (I_m) developed by San Jose *et al*. (1994) (José et al., 1994b) was used according to the equation:

$$I_m = \frac{X_{MNS,Ri}}{X_{MNS,O}} \quad (2)$$

$X_{MNS,Ri}$ is the mass fraction of macadamia nut shells (MNS) for a given bed region (%wt.); and $X_{MNS,O}$ is the initial mass fraction of macadamia nut shells (MNS) in bed (%wt.).

Based on equation (2), the following explanations could be made. Ideal particle mixing was achieved when the mixing index value (I_m) was close to 1. Values of I_m >1 showed a higher concentration of MNS, while those of I_m <1 indicated a higher concentration of sand particles.

2.4. VALIDATION OF MODEL

A new experimental design was used for model validation. The new ranges resulted from the average between the factors listed in Table 2, namely: air velocity (V/V_{MF}; 1.15 and 1.25), particle diameter ratio (D_{MNS}/D_S; 1.18 and 1.64), and MNS mass fraction (X_C; 20 and 40 wt.%).

3. RESULTS AND DISCUSSION

3.1. EFFECT OF FACTORS ON THE MIXING INDEX (I_M)

Table 3 shows the significant effects of the factors (D_{MNS}/D_S, X_C, and V/V_{MF}) on the mixing index (I_m) using a 95% confidence interval, i.e., considering only the effects of factors with a p-value of less than 5%. Among the factors analyzed, only the D_{MNS}/D_S and V/V_{mf} affect the mixing index (I_m). On the other hand, despite V/V_{MF} for the R1 region having a p-value higher than the significant level of 5%, the effect was considered as the parameter was significant for other regions.

Table 3. Estimated effects of factors on I_m for three regions.

Region	Factors	Effect	Standard deviation	t	P-value
R_1	Average	1.02	0.01	96.40	<0.01
	(1) D_{MNS}/D_S	0.08	0.02	4.00	<0.02
	(2) V/V_{MF}	-0.05	0.02	-2.52	<0.07
	1 by 2	-0.09	0.02	-4.47	<0.02
R_2	Average	1.05	0.01	114.85	<0.01
	(1) D_{MNS}/D_S	0.10	0.01	5.71	<0.01
	(2) V/V_{MF}	-0.07	0.01	-3.81	<0.02
	1 by 2	-0.05	0.01	-2.99	<0.05
R_3	Average	0.93	0.01	53.84	<0.01
	(1) D_{MNS}/D_S	-0.19	0.03	-5.45	<0.01
	(2) V/V_{MF}	0.13	0.03	3.73	<0.03
	1 by 2	0.14	0.03	4.16	<0.02

Figure 4, plotted by Chemoface software (Nunes *et al*., 2012), presents the response surface plots for three-bed regions. In general, either higher air velocity or smaller diameter ratio D_{MSN}/D_S favors uniform particle mixing in the bed (bed achieves I_m close to 1); whereas particle segregation is more evident for beds with a larger diameter ratio between MNS and sand ($D_{MNS}/D_S \geq 3$) operating at lower air velocities (V/V_{mf} =1.1).

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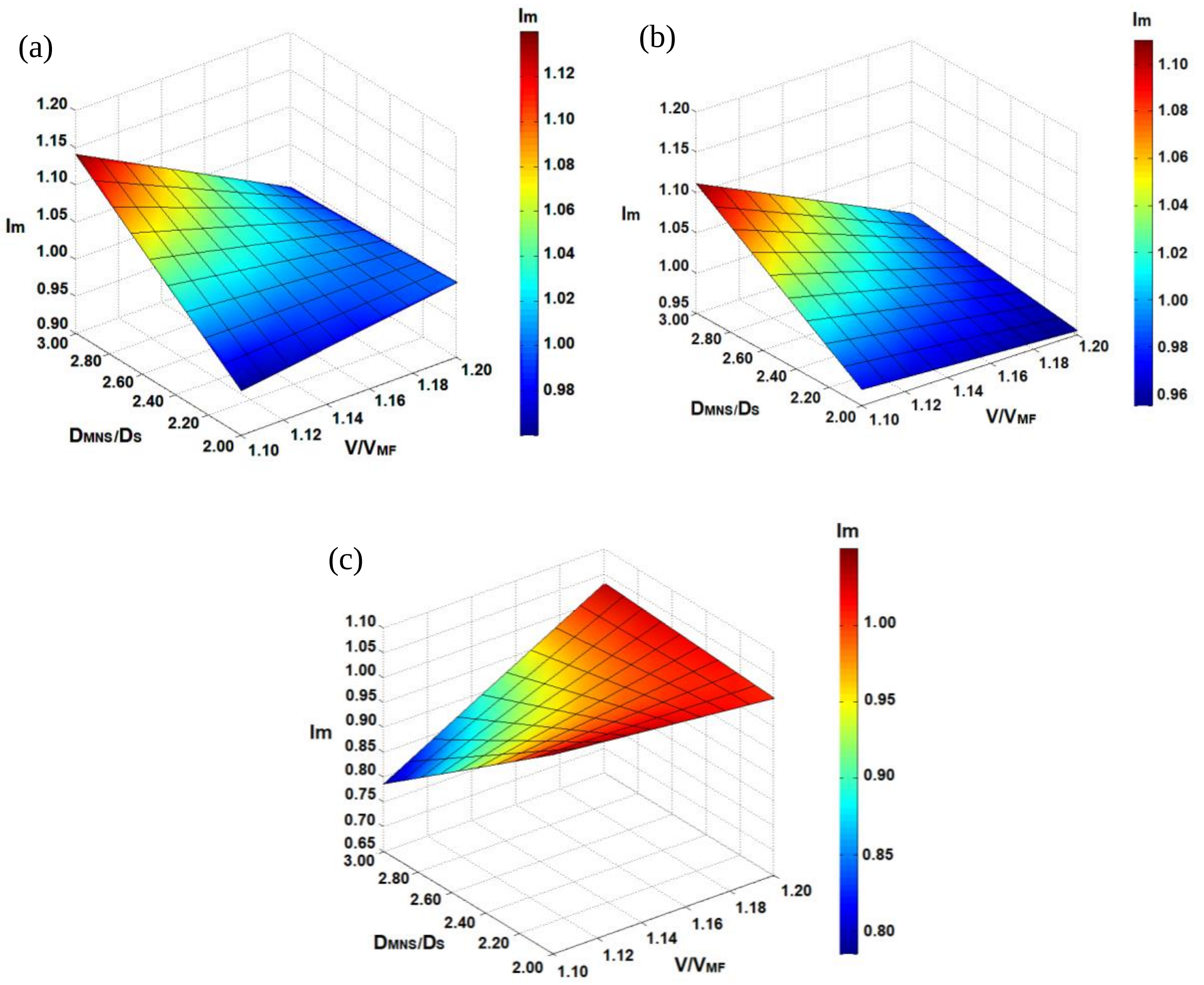
Figure 4. Surface plot for the response variable I_m: (a) R_1 (top), (b) R_2 (medium), and (c) R_3 (bottom).



At the top of the bed (Figure 4-a), particle diameter ratio and air velocity affect the mixing index (Im) mainly due to the action of gravity and drag force. Under the influence of these forces, low-density particles (macadamia nut shells - MNS) are easily entrained and travel longer distances, favoring their accumulation at the top of the bed. A larger MNS particle diameter leads to high thermal and mass resistance lowering the particle heating during the biomass devolatilization in the reactor. Therefore, at an air velocity lower than 20%, MNS and Sand mixtures with a larger diameter ratio ($D_{MNS}/D_S>3$) should be avoided as they would favor particle segregation in the bed, compromising heat transfer and mass transfer rates in the reactor.

At the bottom of the bed (Figure 4-c), the drag force is less effective for the denser particles, i.e., the sand particles, resulting in a concentration of sand particles. This effect weakens when beds with a smaller diameter ratio between macadamia nut shells and sand ($D_{MNS}/D_S < 3$) are used.

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The statistical models predicting the mixing index (I_m) are shown in Table 4. They have a high coefficient of determination (R^2). The residues (not shown herein) are randomly around the zero line and normally distributed, proving the model's adequacy for predicting data tendencies within the limits of the parameters investigated.

Table 4. Model equations for three regions.

Region	Equation	R^2
R_1 (top)	$I_m = -4.02 + 2.27\,(D_{MNS}/D_S) + 4.20\,(V/V_{MF}) - 1.90\,(D_{MNS}/D_S)\,(V/V_{MF})$	0.91
R_2 (medium)	$I_m = -1.56 + 1.37\,(D_{MNS}/D_S) + 2.05\,(V/V_{MF}) - 1.10\,(D_{MNS}/D_S)\,(V/V_{MF})$	0.93
R_3 (bottom)	$I_m = 8.25 - 3.52\,(D_{MNS}/D_S) - 5.90\,(V/V_{MF}) + 2.90\,(D_{MNS}/D_S)\,(V/V_{MF})$	0.94

3.2. VALIDATION OF THE MIXING INDEX (I_M)

The model predicts the I_M based on a new range of parameters for validation, as depicted in Table 5.

Table 5. Mixing index (I_m) and mean absolute error (%E).

		I_m			* E		
D_{MNS}/D_S	V/V_{MJ}	R_1 (top)	R_2 (medium)	R_3 (bottom)	R_1 (top)	R_2 (medium)	R_3 (bottom)
2	1.1	0.94	1.01	1.07	2.00	0.00	3.00
	1.2	1.03	0.99	0.99	3.00	0.50	4.00
	1.1	0.98	1.01	1.01	2.00	0.00	3.00
	1.2	0.97	1.00	1.06	3.00	0.50	3.00
3	1.1	1.12	1.18	0.72	2.00	0.80	2.00
	1.2	0.98	1.01	1.03	1.00	3.00	5.00
	1.1	1.16	1.16	0.69	2.00	0.90	2.00
	1.2	1.00	1.08	0.93	1.00	3.00	5.00

$*\%E = \frac{|Im_{exp} - Im_{model}|}{Im_{exp}} x100$; ** experimental deviations (obtained from replicated data) range from 0.04 to 0.10.

The %E represents a module percental difference between the experimental I_M and the model prediction to the experimental mixing index. Based on data comparisons, the model predicts factors affecting the mixing index (Im) with an %E value of less than 6.00% and is valid for predicting the mixing index for bed mixtures of MSN and sand particles.

4. CONCLUSIONS

Based on the statistical analyses of the experimental design used in this study, the following conclusions can be drawn:

- for all studied bed mixtures, only the diameter ratio (D_{MNS}/D_S) and air velocity ratio (V/V_{MF}) affect the mixing index (Im);

- the segregation of the particles can be avoided if the air velocity ratio (V/V_{MF}) is 20% higher than the minimum fluidization and particle diameter ratio (D_{MNS}/D_S) is lower than 3;

- the model predicts the factors affecting the mixing index (I_m) with an %E of less than 6%.

- the good solid mixing shows a fluidized bed as a suitable reactor for MSN biofuel production.

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As a novelty, this research shows the application of the design of the experiment to access particle concentration along the fluidized bed columns to show particle mixing or segregation impacting the reactor performance in pyrolysis reactor start-up.

For practical applications, the investigated operating conditions (V/V_{MF}>is 20% and D_{MNS}/D_S<3) suggested that the fluidized bed composed of sand and MNS can produce biofuels supporting part of upcoming energy and waste-derived fuel demands.

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