PARTICLE SIZE ANALYSIS OF AGRICULTURAL WASTE RESIDUES AS A PRELIMINARY STEP TOWARDS THEIR VALORIZATION IN BIO-COMPOSITE MATERIALS

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Abstract
Agricultural waste poses both an environmental concern and a valuable resource for sustainable material innovation. Transforming these residues into useful inputs for bio-composites offers a practical path toward waste reduction and eco-friendly development. This study focuses on the particle size characteristics of three agricultural residues—egg shell, corn cob, and fish bone—as an early step toward their potential application in composite materials. The three agricultural waste residues were collected, processed, and ground for sieve analysis. Each sample (50 g) was sieved using mesh sizes from 7 mm to 0.1 mm. Particle weights retained on each sieve were recorded to calculate percentage weight retained, cumulative weight retained, and cumulative percentage passing. Grain size distribution curves were plotted, and robust M-estimators—Huber's, Tukey's Biweight, Hampel's, and Andrews' Wave—were used to identify central tendencies. Normality tests (Kolmogorov-Smirnov and Shapiro-Wilk) showed non-normal distribution, prompting the use of non-parametric tests: Kruskal-Wallis and Jonckheere-Terpstra. Sieve analysis showed that egg shell retained the most particles at 0.1 mm (\[40.278g], \[80.556%]), while fish bone retained broader size ranges, notably at 7 mm (\[1.291g], \[2.582%]). M-estimators revealed Huber's method recorded the highest percentage retention for all residues (\[5.560%] for egg shell). Tests of normality showed only egg shell data deviated significantly (p < \[0.05]). Ranking results indicated fish bone had the highest mean rank in weight retained (\[8.40]), but egg shell ranked highest in cumulative weight passing (\[8.50]). Kruskal-Wallis and Jonckheere-Terpstra tests (p = \[0.949] and \[0.751], respectively) confirmed no significant distribution differences among the residues. These findings are vital for bio-composite design, as particle size directly influences material behavior, bonding quality, surface interaction, and mechanical strength. Understanding these characteristics helps guide the selection and processing of agricultural fillers, ultimately enhancing composite performance and promoting circular economy goals.

1. Introduction

The growing concern over environmental degradation, waste accumulation, and the depletion of non-renewable resources has led researchers to explore sustainable alternatives in material science. One such approach is the valorization of agricultural waste residues into bio-composite materials. The first necessary part of the valorization process is to measure the particle size, as this directly affects the physical, mechanical and chemical behavior of the composites (Okafor et al., 2022a). Often, waste from agriculture, for example, rice husk, coconut shell, corn stalks, groundnut husks and plantain peels, ends up being burned in the open or allowed to rot, causing harm to nature and wasting useful biomass. Transforming these waste materials into bionic composites could be valuable, but there are still difficulties in making sure they are applied consistently. Having different sized particles in the agricultural residue can prevent proper compatibility, dispersion and bonding within the polymer matrix (Sholokhova et al., 2025; Ihueze et al., 2017). If the distribution and control of particle sizes are not strong, bio-composites will not perform mechanically or thermally well and will lose efficiency.

Before processing lignocellulosic agricultural residue for composites, particles need to be analyzed for size. This analysis shows how the size and number of particles influence the amount of surface area and the interaction between the filler and the surrounding material (Agrawal et al., 2021; Okafor et al., 2024). Finer and uniformly spread particles are usually selected to provide good mechanical bonding and smooth surfaces in composite structures. The works of Okafor et al. (2022b) and Abdelmagid et al. (2024) suggest that the tensile strength and water absorption in natural fiber composites depend on the particle size. The study by Adeleke et al. (2023) on composite materials made from carbonized cassava back peel and iron fillings found that particles smaller in size resulted in materials with higher tensile strength and less water absorption due to good compaction and reduced voids. Darekar et al. (2024) also conducted studies on particle size analysis for effective reinforcement in epoxy-based composites used in electronics. It was the increase in contact between the filler and the epoxy that made the composite stronger. The data clearly points out that particle size plays a key role in achieving desired composite properties.

In another investigation, Mandala et al. (2023) reviewed the mechanical features of peanut-shell-polymer composites. The investigation revealed that finer particles play a bigger role in decreasing the strength of the material, with coarser particles creating more points where the material could fail because of low interfacial adhesion. The study proposes sieving and grinding methods to uniform the size of particles before using them in composites. Additionally, particle size has a significant influence on the conduct of heat and the stability of particles. Kannan and Thangaraju (2023) reported that when plantain stem waste is ground into smaller pieces, it boosts the polyester composites' ability to withstand heat. According to the findings, raising the packing density helped to block air gaps in the composites. The size of the particles plays a role in determining the viscosity and handling characteristics of the mixture while making composites. If the particles are not well optimized, they might prevent the mixture from being mixed evenly, creating a composite with poor properties (as mentioned by Huang et al in 2015). Thus, grinding, filtering and separating particles is necessary to create a uniform size and to make them suitable for incorporation into the polymer matrix.

There is a growing reason for this study because agricultural waste needs to be used to help the environment. The fact that bio-composite development can use these wastes is well known, but scientists pay little attention to particle size during research, focusing more on chemicals and compatibility with matrices (Szadkowski et al., 2025). Particle size determines the distribution of filler, the bonding strength of materials and how well the composite performs (Elfakhri et al., 2022). However, gaps remain in standardized methodologies for particle size optimization across various agro-residues. Without precise particle size analysis, the mechanical and thermal properties of resulting composites can be inconsistent. This study is therefore motivated by the need to bridge this gap by establishing particle size analysis as a critical pre-treatment step in the valorization of agricultural wastes for enhanced bio-composite performance.

2. Method

Samples of three agricultural waste residues: egg shell, corn cob, and fish bone were first collected from local processing sites. Egg shells were washed to remove any attached membrane or organic residue and then sun-dried. Corn cobs were also dried after kernel removal, while fish bones were boiled to eliminate flesh and fat before being sun-dried. All the samples were ground using a laboratory mill to reduce them to fine particles suitable for sieve analysis. For consistency and comparability, 50 grams of each ground residue were measured for the particle size tests. Figure 1 outlines the particle size analysis workflow. Each material underwent mechanical grinding using a blender and sieving through a mechanical shaker to obtain uniform particle sizes.

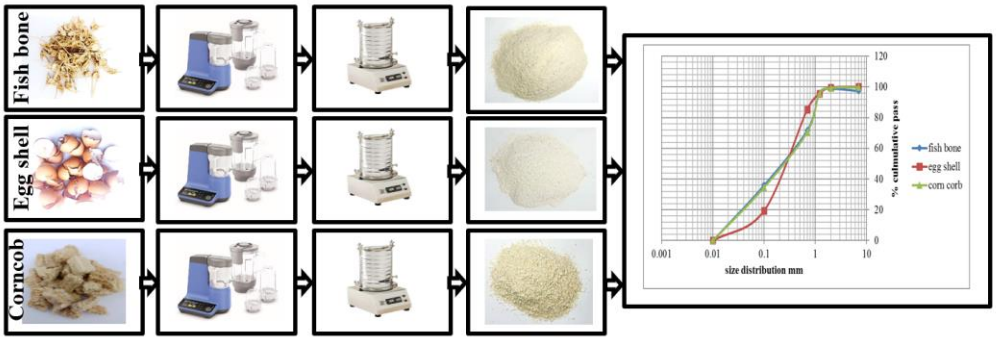

Figure 1. Particle Size Analysis Workflow for Three Agricultural Waste Residues

Sieve analysis was carried out using a mechanical shaker and a standard set of laboratory sieves with mesh sizes ranging from 7 mm to 0.1 mm. The 50-gram sample was put in the top sieve and mixed by mechanical means to allow the particles to divide by size. After screening the sand with sieves, what remained on each one was weighed down and the values recorded. Key indices like percentage retained, the total weight kept and cumulative percentage passing were figured out using the weights for every type of residue. I used this approach because it can accurately display the types of particles in bulk materials which shows if the remaining material can be used again or processed industrially.

Grain size distribution curves were created using the sieve measurements to display the way particle sizes were spread in each sample. Robust M-estimators were used to analyze the particle weight data for better understanding. Among them were Huber's, Tukey's Biweight, Hampel's and Andrew's Wave estimators. These tools made it possible to identify central values in measurements by not letting extreme ones affect the result. Huber's estimate aimed for a position between the mean and the median, whereas Tukey's Biweight and Andrews' Wave were developed to suppress extreme values and highlight moderate ones. Managing the wide range of sizes among the titanium particles was eased by the fact that Hampel's estimator is adaptively robust, especially helpful when materials like fish bone were analyzed.

Tests for normality were conducted using both the Kolmogorov-Smirnov and Shapiro-Wilk tests. Due to the observed non-normal distribution, particularly in the eggshell sample, the study employed non-parametric statistical methods to assess whether differences existed in particle size characteristics among the three residue types. The Kruskal-Wallis test was used to compare the mean ranks of weight distributions across eggshell, corn cob, and fish bone. The Jonckheere-Terpstra test was also applied to check for an ordered trend in the ranks across the three materials.

3. Results and Discussion

Table 1. Particle Size Distribution of Agricultural Waste Residues Based on Sieve Analysis (Sample Weight = 50g)

Agricultural waste residue	Sizes				
		Weight g	% weight retained	% cumul wt retained	% cumul wt passing
	0.1	40.278	80.556	80.556	19.444
Egg shell	0.7	7.278	14.556	14.556	85.444
	1.2	2.186	4.372	4.372	95.628
	2	0.258	0.516	0.516	99.484
	7	0.00	0	0	100
Corn corb	0.1	32.676	65.352	65.352	34.648
	0.7	14.676	29.352	29.352	70.648
	1.2	2.178	4.356	4.356	95.644
	2	0.334	0.668	0.668	99.332
	7	0.136	0.272	0.272	99.728
Fish bone	0.1	32.022	64.044	64.044	35.956
	0.7	14.116	28.232	28.232	71.768
	1.2	2.006	4.012	4.012	95.988
	2	0.565	1.13	1.13	98.87
	7	1.291	2.582	2.582	97.418

Sieve analysis of egg shell, corn cob, and fish bone as presented in Table 1 reveals how particle sizes affect weight retention and passing. Egg shell retained the most weight at 0.1 mm ([40.278g], [80.556%]), with a sharp drop across larger sizes, reaching 100% passing at 7 mm. Corn cob followed a similar trend, retaining ([32.676g], [65.352%]) at 0.1 mm and tapering off with minimal retention beyond 1.2 mm. Fish bone had ([32.022g], [64.044%]) at 0.1 mm but retained more at larger sizes (e.g., [1.291g], [2.582%] at 7 mm). Egg shell disintegrated more finely, while fish bone displayed broader size retention across the sieves.

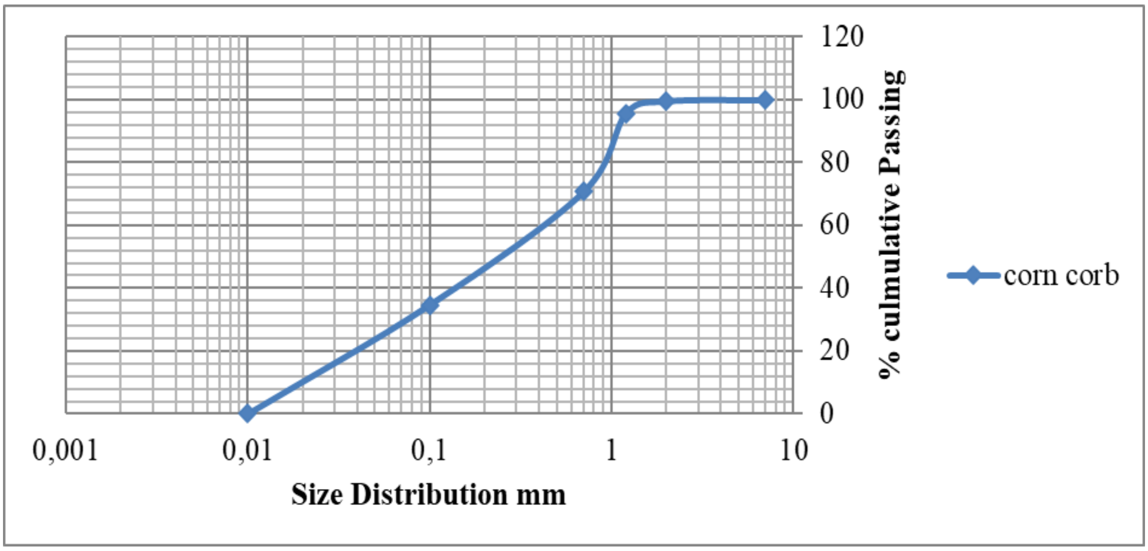

Figure 2. Grain Size Distribution Curve for Corn Cob Residue

The graph in Figure 2 shows the cumulative percentage passing of corn cob particles across sieve sizes. A sharp rise is observed between 0.1 mm and 1.2 mm, with cumulative passing increasing from ([34.648%]) at 0.1 mm to ([99.728%]) at 7 mm. The curve indicates a well-graded distribution, with most particles finer than 1.2 mm, suggesting suitability for fine processing applications.

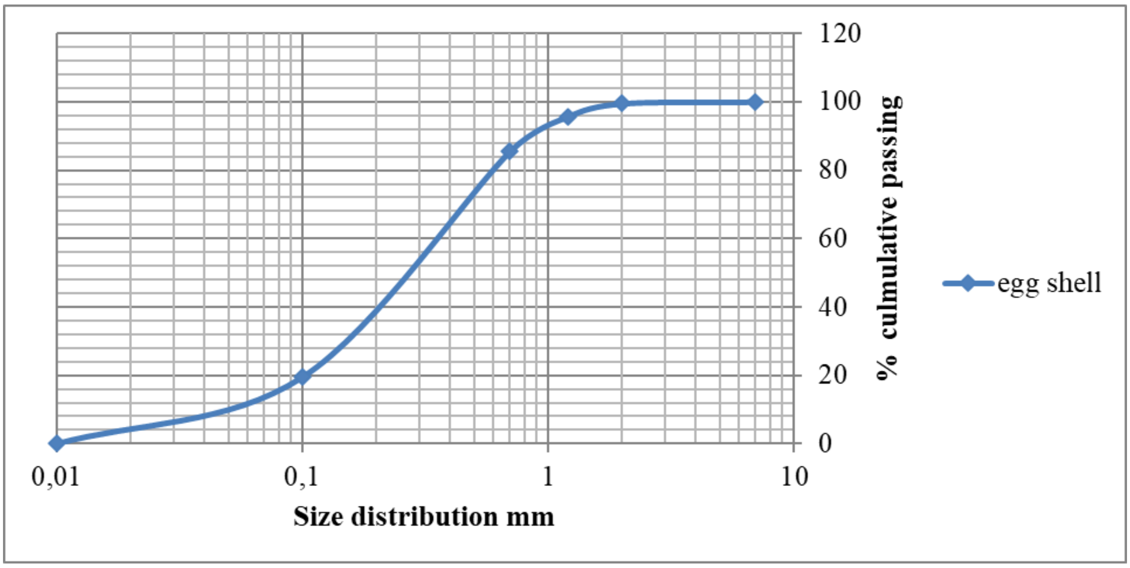

Figure 3. Grain Size Distribution Curve for Egg Shell Residue

The curve in Figure 3 illustrates the cumulative passing of egg shell particles across sieve sizes. A steep increase is observed from ([19.444%]) at 0.1 mm to ([95.628%]) at 1.2 mm, reaching ([100%]) at 7 mm. The distribution shows finer particles dominate, indicating the egg shell residue is highly friable and suitable for fine powder applications in composite or agricultural use.

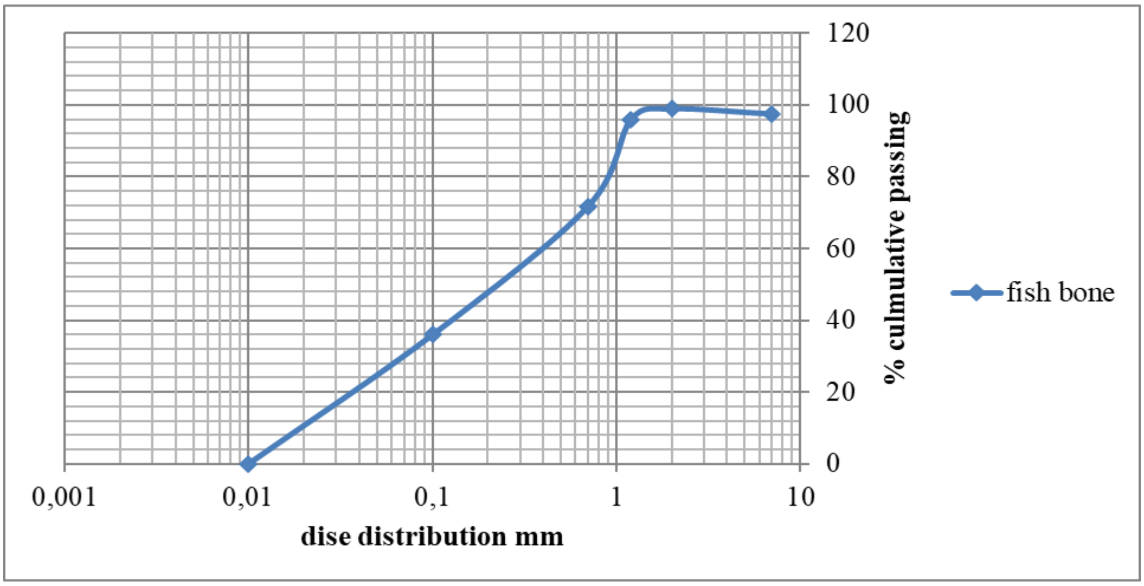

Figure 4. Grain Size Distribution Curve for Fish Bone Residue

The graph in Figure 4 reveals that fish bone residue exhibits a gradual increase in cumulative passing from ([35.956%]) at 0.1 mm to ([95.988%]) at 1.2 mm, reaching a maximum of ([97.418%]) at 7 mm. This distribution indicates a wide range of particle sizes with a dominance of finer particles, making it suitable for use as filler material in composite production.

Table 2. Comparison of M-Estimators on Particle Weight Distribution of Agricultural Waste Residues

	Agricultural Waste Residue	Huber's M-Estimator[a]	Tukey's Biweight[b]	Hampel's M-Estimator[c]	Andrews' Wave[d]
Percentage Weight Retained	Egg shell	5.560	3.640	4.129	3.648
	Corn corb	5.459	1.776	2.707	1.775
	Fish bone	5.233	2.567	2.567	2.568
Percentage Cumulative Weight Retained	Egg shell	5.560	3.640	4.129	3.648
	Corn corb	5.459	1.776	2.707	1.775
	Fish bone	5.233	2.567	2.567	2.568
Percentage Cumulative Weight Passing	Egg shell	94.479	96.296	95.820	96.289
	Corn corb	94.550	98.221	97.258	98.221
	Fish bone	94.821	97.431	97.433	97.431

a. The weighting constant is 1.339.
b. The weighting constant is 4.685.
c. The weighting constants are 1.700, 3.400, and 8.500
d. The weighting constant is 1.340*pi.

Table 2 presents percentage weight retained and cumulative weight retained/passing for egg shell, corn cob, and fish bone across four M-estimators. Huber's estimator showed the highest weight retention for egg shell (5.560%) and corn cob (5.459%), while fish bone was highest under Huber's and Hampel's (5.233%). Tukey's and Andrews' produced lower weight retention, especially for corn cob (1.776%, 1.775%). Cumulative weight passing was highest under Tukey's for corn cob (98.221%) and fish bone (97.433%). Huber's estimator retained the most mass, while Tukey's and Andrews' favored finer particle distributions, indicated by higher weight passing percentages.

Table 3. Results of Kolmogorov-Smirnov and Shapiro-Wilk Tests of Normality

	Agricultural Waste Residue	Kolmogorov-Smirnov[a] Statistic	df	Sig.	Shapiro-Wilk Statistic	df	Sig.
Percentage Weight Retained	Egg shell	.363	5	.030	.687	5	.007
	Corn corb	.311	5	.128	.799	5	.079
	Fish bone	.323	5	.096	.789	5	.065
Percentage Cumulative Weight Retained	Egg shell	.363	5	.030	.687	5	.007
	Corn corb	.311	5	.128	.799	5	.079
	Fish bone	.323	5	.096	.789	5	.065
Percentage Cumulative Weight Passing	Egg shell	.363	5	.030	.687	5	.007
	Corn corb	.311	5	.128	.799	5	.079
	Fish bone	.323	5	.096	.789	5	.065

a. Lilliefors Significance Correction

The Tests of Normality using both Kolmogorov-Smirnov and Shapiro-Wilk in Table 3 indicate that the distribution of data for Egg shell is not normally distributed, as its p-values (Sig.) are below 0.05 for all variables: percentage weight retained (.030/.007), cumulative weight retained (.030/.007), and cumulative weight passing (.030/.007). In contrast, Corn corb and Fish bone show p-values greater than 0.05 (Corn corb: .128/.079; Fish bone: .096/.065), suggesting that their distributions are approximately normal. Based on this, non-parametric tests such as the Kruskal-Wallis test was considered when including eggshell in group comparisons due to its deviation from normality.

Table 4. Mean Ranks of Agricultural Waste Residues across Particle Weight Distribution Parameters

	Agricultural Waste Residue	N	Mean Rank
Percentage Weight Retained	Egg shell	5	7.50
	Corn corb	5	8.10
	Fish bone	5	8.40
	Total	15	
Percentage Cumulative Weight Retained	Egg shell	5	7.50
	Corn corb	5	8.10
	Fish bone	5	8.40
	Total	15	
Percentage Cumulative Weight Passing	Egg shell	5	8.50
	Corn corb	5	7.90
	Fish bone	5	7.60
	Total	15	

The ranking analysis in Table 4 compares egg shell, corn cob, and fish bone based on mean ranks for weight distribution. For both percentage weight retained and cumulative weight retained, fish bone had the highest mean rank ([8.40]), followed by corn cob ([8.10]) and egg shell ([7.50]). In contrast, for cumulative weight passing, egg shell ranked highest ([8.50]), while corn cob ([7.90]) and fish bone ([7.60]) followed. These results suggest fish bone tends to retain more weight, indicating coarser particles, while egg shell allows more weight to pass through, reflecting finer particle size. The total number of observations per group was consistent ([N = 5]).

Table 5. Kruskal-Wallis Test Statistics for Particle Weight Distribution across Agricultural Waste Residues

	Percentage Weight Retained	Percentage Cumulative Weight Retained	Percentage Cumulative Weight Passing
Chi-Square	.105	.105	.105
df	2	2	2
Asymp. Sig.	.949	.949	.949

a. Kruskal Wallis Test
b. Grouping Variable: Agricultural Waste Residue

The Kruskal-Wallis test in Table 5 assessed differences in particle weight distribution among egg shell, corn cob, and fish bone. Chi-square values were low across all parameters—percentage weight retained ([0.105]), cumulative weight retained ([0.105]), and cumulative weight passing ([0.105])—with degrees of freedom ([df = 2]). The asymptotic significance values were high ([0.949] for all), indicating no statistically significant differences among the agricultural residues in any of the measured parameters. These findings suggest that the particle weight characteristics are similar across the three residue types, with variations in rankings not being strong enough to denote significant differences in their distribution patterns.

Table 6. Jonckheere-Terpstra Test Results for Ordered Differences Among Agricultural Waste Residues

	Percentage Weight Retained	Percentage Cumulative Weight Retained	Percentage Cumulative Weight Passing
Number of Levels in Agricultural Waste Residue	3	3	3
N	15	15	15
Observed J-T Statistic	40.500	40.500	34.500
Mean J-T Statistic	37.500	37.500	37.500
Std. Deviation of J-T Statistic	9.455	9.455	9.455
Std. J-T Statistic	.317	.317	-.317
Asymp. Sig. (2-tailed)	.751	.751	.751

a. Grouping Variable: Agricultural Waste Residue

The Jonckheere-Terpstra test in Table 6 examined ordered trends across egg shell, corn cob, and fish bone for weight retention and passing. For all three parameters—percentage weight retained, cumulative weight retained, and cumulative weight passing—the observed J-T statistics were ([40.500]), ([40.500]), and ([34.500]) respectively, compared to a mean J-T of ([37.500]) with a standard deviation of ([9.455]). The standardized J-T values were low ([0.317], [0.317], and [-0.317]), and asymptotic significance was non-significant across all tests ([0.751]). These results indicate no significant monotonic trend among the waste types, suggesting that the differences in weight behavior are not directionally ordered across the residues.

Particle size analysis of agricultural waste residues such as egg shell, corn cob, and fish bone play a pivotal role in determining their suitability for incorporation into bio-composite materials. As shown in Table 1 and Figures 2 to 4, sieve analysis revealed distinct particle size distributions among the residues. Egg shell exhibited the finest particles, with 80.556% retained at 0.1 mm and a complete 100% passing at 7 mm, indicating high friability. In contrast, fish bone demonstrated a broader distribution, with 2.582% retained at 7 mm, suggesting a coarser structure. Corn cob showed intermediate behavior, making it suitable for structural and filler roles in bio-composite formulations. This finding agreed with Kumar and Saha (2022), who emphasized that finer agricultural particles enhance the interfacial bonding between fillers and polymer matrices in composite materials, thereby improving mechanical strength and thermal stability. In a related study, Okafor et al, (2022b) confirmed that the particle size distribution directly influences load transfer efficiency and matrix-filler adhesion in natural fiber composites. The friable nature of egg shell implies high surface area, a property desirable for chemical interaction within bio-resin systems (Owuamanam & Cree, 2020).

In contrast, fish bone's broader distribution, as evident in its retention at larger sieve sizes, aligns with the report by Agrawal et al, (2021), which found that coarser fillers contribute better to structural integrity but may compromise homogeneity. Similarly, the well-graded profile of corn cob as depicted in Figure 2 supports its use in load-bearing applications. This finding supported Kumar and Saha (2022), who identified corn cob as a cost-effective reinforcement in biodegradable composites, especially where moderate stiffness and density are desired. Table 2 reveals that Huber's M-estimator yielded the highest weight retention across all residues, indicating robustness in measuring mass distribution. In contrast, Tukey's Biweight and Andrews' Wave estimators favored fine particles, with egg shell showing the highest cumulative passing (96.296%). This trend corroborates the assertion of Zhang et al (2022), who found that finer fractions lead to improved dispersion in polymer matrices, especially when using non-parametric M-estimators for statistical modeling of composite ingredients.

However, the Shapiro-Wilk and Kolmogorov-Smirnov tests in Table 3 show a deviation from normality in egg shell data ($p < 0.05$), unlike corn cob and fish bone. This result aligns with the study of Li et al (2015), which emphasized the importance of selecting appropriate statistical models when dealing with non-normally distributed data. Consequently, non-parametric tests like Kruskal-Wallis were adopted for further analysis. Interestingly, Table 4 shows fish bone having the highest mean rank for weight retention, suggesting its comparative coarseness. In contrast, egg shell dominated in cumulative weight passing. These outcomes align with the findings of Ali et al (2022), who demonstrated that fine powders enhance aesthetic and surface properties, whereas coarser particles serve better as structural reinforcements in hybrid composites.

The insignificance observed in Table 5 from the Kruskal-Wallis test (p = 0.949 across all variables) suggests homogeneity in weight distribution among the residues. In contrast, findings from Malley et al (2022) reported significant variations in mechanical outputs of composites when particle distributions differ substantially. As a result, the similarities in particle weight make it easy to blend these residues with other materials, allowing for better composite results. Particle size analysis is important because it affects parameters such as strength, durability and the way materials are processed. Similarly, this work agrees with Okafor et al, (2021), who discovered that using finer particles improved wetting and decreased void content when forming banana-coir composite parts. In a further study, Okafor et al, (2022b) and Okafor et al, (2018) found that having graded particles in wood residues leads to improved mechanical behavior because energy is dissipated more effectively. The study reveals that egg shell is fit for fine uses including bio-fillers or nano-composites, compared to corn cob and fish bone for structural purposes.

4. Conclusion

The particle size analysis of selected agricultural waste residues provides critical foundational understanding into their potential application in bio-composite material production. This study established that the physical characteristics—particularly particle size distribution which plays a significant role in determining the compatibility, interfacial bonding, and mechanical performance of bio-composites. The assessment revealed that residue particle sizes varied which suggests that suitable uses for such material would depend on its size. Uniformly small particles tend to provide a more even and strong structure in composites, but coarse particles are better for filling roles that do not require much strength. When these factors are understood, the selection and processing of waste from farms become easier, with less negative impact on the environment and greater focus on sustainability. This first analysis points out that additional research is needed to study the chemical properties and functional use of bio-composites using these materials. Reusing agricultural waste in bio-composites endorses a circular economy and gives industries an environmentally friendly and economic alternative to synthetic fillers.

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