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Bio-composite Material Characterization Based on Field Emission Scanning Electron Microscopy Images

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Abstract

Measuring the texture properties of the sample surface is an important matter for bio composite materials. In this research, the samples were prepared from egg shells and palm fronds, and a set of pictures were taken in the Field Emission Scanning Electron Microscopy (FESEM) of many samples before and after hydrochloric acid immersion. Then, the image processing and analysis phase begins by taking images of different sizes and measuring the texture properties of the images. The statistical measurement results were performed on the samples to specify their effect on the acid solution. Many factors were applied, including; contrast, dissimilarity, homogeneity, angular second moment, maximum probability, entropy, energy, mean, variance, and correlation. After studying and comparing the samples' results and examining the acid effect on them, it was found that the two samples are resistant to acid immersion; the palm frond sample is more resistant to acid immersion than the eggshell sample. The results are quite acceptable compared with the previous studies.

Keywords: *Egg Shells composite material, FESEM morphology, Palm Fronds Composite Material, Texture Measures.*

1 Introduction

1.1 Composite Material

Two types of natural composite material are used in this work:

- **Palm Fronds (PF)**

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The date palm tree, a member of the Phoenix Dactylifera palm tree family, is commonly found in California, the Canary Islands, Northern Africa, India, Pakistan, and Iraq [1]. Over 16 million date palms grow in Iraq. Every year, it produces around 630,000 tons of date palm remnants [1]. Table 1: Chemical composition of different portions of date palm tree fronds [2].

Table 1: Chemical components of various portions of date palm tree fronds.

	Cellulose	Hemicelluloses	Lignin	Ash	Extractive
Leaflet	40.21	12.8	32.2	10.54	4.25
Rachis	38.26	28.17	22.53	5.96	5.08

- **Egg Shell (ES)**

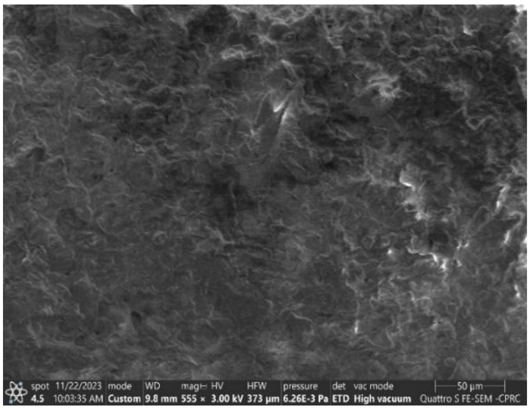
The tough exterior coating of an egg, known as the chicken egg shell (ES), is available in brown or white colors [3. A chicken egg's weight composition is 60% albumen, a white substance, 30% yolk, a yellow substance, and 10%-11% shell, which includes the eggshell and membrane [4]. An egg is said to weigh between 60.0 and 60.2 grams, while an empty shell weighs between 6.6 and 7.3 grams [5, 6]. The shell contains a small amount of organic materials, estimated to be between 3 and 4.5 weight percent [7, 8]. Various examinations have discovered that the density of the ES together with the membrane ranges from 2.50 to 2.62 g/cm3, and the membrane's density was 1.36 g/cm3 [9,10]. The majority of egg shell's (ES) chemical makeup is made up of CaCO3 in the form of calcite, which makes up between 94 and 98 weight percent [9]. There are also trace amounts of other elements including magnesium and phosphorus as well as 3–4% organic content [9, 10]. Table 2.2 provides a summary of certain CaCO3 contents that have been reported [11]. The composition of ES can be affected by inner membrane pollution and the chicken's diet [12]. Based on the research, brown ES includes 96–97 weight percent CaCO3 and 3-4% organic content. White ES had 94 weight percent CaCO3 and 6 weight percent organic materials [13]. According to recorded results, the CaCO3 concentrations in brown and white ES were determined to be comparable [14].

Table 2: CaCO3 composition of eggshell

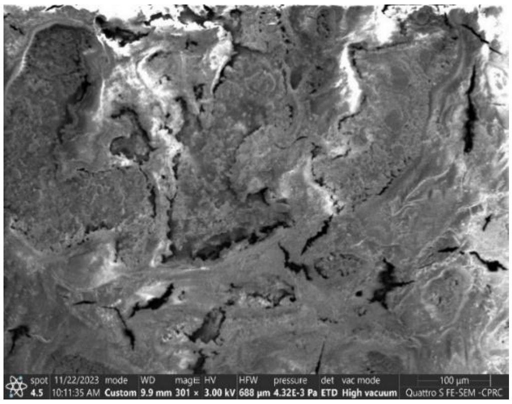
$CaCO^3$ content (wt. %)	Eggshell density (g/cm^3)	References
94.00	2.53	[13]
96.41	2.59	[14]
97.30	2.62	[15]
97.80	2.50	[11]

1.2 FESEM Images

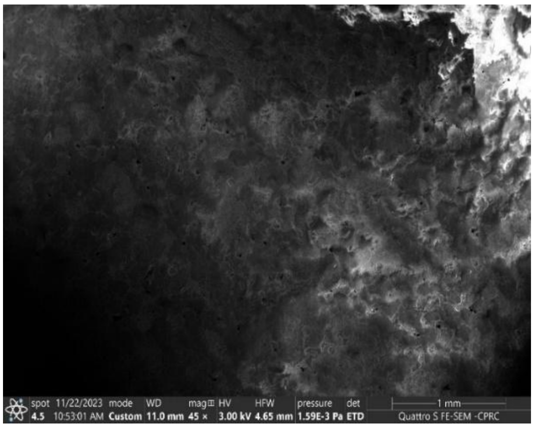
Field emission scanning electron microscopy (FESEM) was accomplished to test the morphology of the arranged composite materials. The field emission scanning electron microscopy type (Thermoscientific Quattro S) was utilized to explore the arranged samples. The FESEM morphology images captured from the sample surface of treated and untreated composite materials with hydrochloric acid (HCl) are shown in Fig. 1. The gained FESEM images demonstrated that the sample surfaces comprise two overlapping regions. These regions are the smooth and rough regions. The smooth region is the epoxy matrix, while the rough region is attributed to the added powders (ES powder and PF powder), respectively. Fig. 1(a-d) demonstrated that there is no apparent effect on the surface of samples after the hydrochloric acid treatment when comparing the FESEM images (a and b) and (c and d) of untreated and treated Ep/ES and Ep/PF samples.



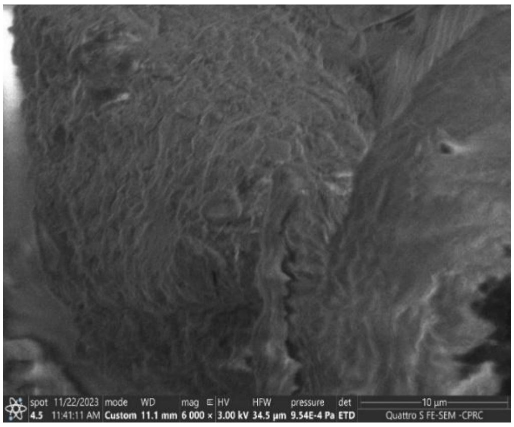
(a) Untreated Ep/ES



(b) Treated Ep/ES with HCl



(c) Untreated Ep/PP



(d) Treated Ep/PF with HCl

Figure 1. FESEM morphology images of prepared composite materials

The characterization of bio-composite materials has obtained great attention owing to their potential applications in sustainable engineering and biomedical fields. The utilization of Field Emission Scanning Electron Microscopy (FESEM) for detailed morphological characterization has not been discovered yet, particularly in the context of bio-composites. Available studies primarily concentrate on conventional microscopy methods, missing a comprehensive technique that leverages FESEM's high-resolution capabilities. This study aims to systematically analyze the microstructural properties of bio-composite materials using FESEM, supplying visions into surface morphology. The study aims to enhance understanding of bio-composite behavior.

2 Related Work

Muller H.G., characterized texture as a combination of physical characteristics fundamentally seen through touch, which can be measured impartially utilizing mass, time, and removal. He recommended two catchphrases to replace the word surface: haptaesthesis, a teaching of physiology that refers to how materials carry on mechanically, and rheology, a portion of physics that portrays the characteristics of food. Rheology considers the behavior of bodies with at least one of the properties of flexibility, thickness, or plasticity [15, 35]. Hameed et al., provided intuition about the use of digital image processing in the field of composite material reinforcement and detection. Digital image processing becomes an effective tool to obtain details of material surface enhancement. The tasks in this work are performed to perform numerically to reach the main goal of the work such as feature extraction and classification process. Several polymer mixtures with different concentrations (4%, 6%, and 8%) were prepared from polycarbonate to test the system. Comparison between the images before and after processing shows a little difference between the images, the processing image gives better focus and on the other hand, the thickness of the model has a small effect on the surface aspect [16,34]. Hameed et al. found that the thermal accessibility of micro, nano, and hybrid alumina and titanium (EP/Al2O3 + EP/TiO2) increases with increasing weight fractions before immersion in HCl. The thermal conductivity decreases with the increase in the value of the weight fraction of TiO2 in both microparticles and nanoparticles, while after immersion in chemical solutions (HCl), thermal conductivity increases with the immersion in chemical solutions. The composite (EP/TiO2) indicated the optimal value of thermal accessibility at a weight ratio of 0.08 and the lowest value of thermal accessibility was displayed at a weight ratio of 0.02 [17]. Hameed et al. focused on applying several image processing functions to understand and analyze the surface properties of composite materials (nano-alumina and nano-titanium). This approach included several steps: enhancement, surface analysis, edge detection, and contour analysis. This approach extracted and evaluated the surface texture properties of the composite material by measuring the average values of the original gray image, the modified image, the equalized image, and the adapted image. Finally, the applied method for texture analysis indicated a significant improvement in surface properties for both the equalization and adaptation methods compared to the modification method for both samples [18, 33]. Armi et al. talked about the different strategies utilized for surface or investigation in detail. This paper presented a survey of well-known combinational strategies in a particular area with subtle elements. It also checks the points of interest and drawbacks of well-known surface picture descriptors within the result portion. The most center in all of the surviving strategies is on separation execution, computational complexity, and resistance to challenges such as commotion, and revolution [19]. Hameed et al. carried out this approach in several stages: material preparation, molding of samples, obtaining a surface image, enhancing the surface image, and measuring several parameters to ensure improvement. Median, Gaussian, mean, and standard deviation filters are applied to the surface image to realize this approach. The acquired results from the standard deviation filter achieved high performance compared to other filters, which have high PSNR values (54) and low MSE values (0.25). Image analysis tools produced good results for texture surface analysis. This is specified in the image enhancement procedures [20]. Espinasse M et al., showed texture analysis in medical imaging could be a promising apparatus that's outlined to progress the characterization of unusual pictures from patients, to eventually serve as a prescient or prognostic biomarker. Within the area of image processing, texture highlights are crucial visual prompts with complemental parts. They are utilized in numerous applications and a huge assortment of regions, like quality control, content-based image recovery, inaccessible detecting, mechanical review, surface assessment, object recognition, and medical image analysis [21]. Ramola et al. displayed a point-by-point study of four distinctive techniques that have been created for texture classification. These strategies incorporate a gray-level co-occurrence matrix, local binary pattern, autocorrelation work, and histogram pattern. The detailed examination of these strategies recommends that gray-level concurrence is best for analyzing surface texture, land-use classification, and satellite data processing [22]. Li X. proposed a class of image texture analysis calculations based on anisotropic dissemination equations and discrete gray-level sets. To begin with, a class of nonconvex generalized capacities was proposed to expel the noise from the initial picture to get a smooth image while honing the edges. At that point, a vitality generalization work depend on the gray level set was suggested, and the presence of the worldwide least of this vitality generalization work was examined. Lastly, a comparable shape of this energy generalization is given within the discrete case, and an image surface investigation calculation is planned based on the proportionate shape [23]. Hameed et al. examined the impact of exposure to gamma rays on magnesium oxide nanoparticles (Nano MgO). Different weights of percentages 2%, 4%, 6%, and 8% were used to prepare the molds. Several steps of the image processing process were applied for texture analysis. The standard deviation, range, and entropy values were measured before and after exposure to gamma rays. The gained results indicated that samples with the percentage of weights of 2% and 4% were more affected by exposure to gamma rays. On the other hand, samples with percentage weights of 6% and 8% were least affected by exposure to gamma rays [24]. Ghalati et al. presented an overhauled overview of texture analysis strategies. As a comprehensive overview, the peruser was presented with an amplified and granular categorization of the texture analysis strategies covering diverse angles and patterns within the field. An intensive survey of handcrafted texture analysis strategies was specified, covering both classical techniques and developing categories. The learning-based techniques in texture investigation cover deep-learning forms and indicate the utilization of high-performance CNNs in texture analysis. Also discussed is the ability of texture analysis in biomedical images [25]. Humeau-Heurtier presented a comprehensive study of the texture, including extraction strategies distributed for color pictures. He partitioned the strategies into seven classes. Two of them are exceptionally later: within the scope of the handcrafted methods, the foremost later course for surface highlight extraction is the one that depends on entropy measures. These strategies have the benefit of depending on popular unidimensional entropy measures. In addition, they can be related to multiscale approaches to thinking about the surface at distinctive spatial scales [26]. Roy et al. proposed a study in which different picture texture analysis strategies are assessed to recognize a solid surface pointer. Strategies incorporate conventional measurable approaches such as the histogram strategy, Grey Level Co-occurrence Matrix strategies, and transform-based wavelet texture analysis [27]. Olejnik et al., assessed the appropriateness of the texture analysis to evaluate the consistency of fluid entrance through the paper structure in terms of the degree of the paper measuring. They displayed a modern strategy of measuring based on energetic changes in their texture parameters [28]. Gibson R.K. et al. explained that the texture is a natural property of all arrival cover surfaces that are known to differ between fire seriousness classes, getting progressively more homogenous as fire seriousness increases. They compared candidate backscatter and reflectance records determined from Sentinel 1 and Sentinel 2, separately, alongside grey-level-co-occurrence-matrix inferred texture indices employing an irregular timberland administered classification system. Cross-validation and target-trained models were compared to assess execution between the models with and without texture files [29]. It is a challenge to recognize the texture even for the human eye [30, 31, 32, 36, 37].

3 Methodology

The methodology in this approach was divided into three stages as shown in Fig. 2:

1-Sample formation stage: this includes grinding the samples, mixing them, preparing the mold, pouring the sample and leaving it to dry, and then immersing the sample in the hydrochloric acid solution.

2-Image preparation stage: this includes taking photos using field emission scanning electron microscopy before and after acid immersion.

3-Image analysis stage: It includes taking samples of the images and applying many factors and functions to them to discover the extent of the change that occurs in them after immersion in the acid solution.

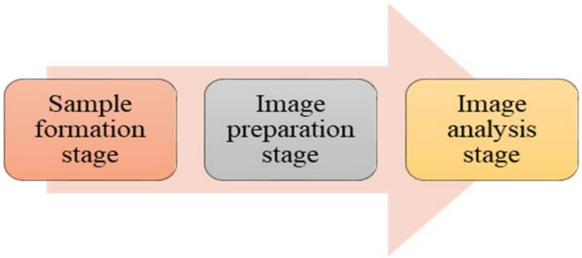


Figure 2. Methodology stages

3.1 Powders Preparation

Two types of powders are prepared. The first one is that the palm fronds utilized as fillers were gained from the date palm tree from the annual pruning of the date palm (Al-Khastawi variety, Iraq), and its grain size was 50µm. Date palm pruning residues were left in the air for 3 months to reach a balanced moisture content and then ground using a shredder, and then the particles were ground with a small laboratory mill followed by passing through a (50µm) sieve and preserved on a cloth. They are then oven-dried at 100 ± 5 °C for 24 hours to repel moisture and stored in airtight plastic bags before installation. The other one is eggshells used as fillers, gained from the eggs of chickens accessible in the social environment. They are oven-dried at 100°C for an hour to repel moisture. It is then ground utilizing the grinding machine dedicated to grinding solids, then passed through a 50-micron sieve. Eggshell powder with a grain size of (50µm) microns. Then they are placed in sealed plastic bags to keep them from moisture.

3.2 Samples Preparation

Samples are prepared by hand, and this method includes the following:

1- Preparing the form: The use of two special molds that include the casting process and the base of each glass plate is coated with a thermal paper mold (to prevent the resin from sticking to the glass plate and facilitate the manufacture of the cutting product) with a high degree of objective plates (ensuring the leveling balance by the flowing surface). The sides of the two molds are composed of different glass ruler thicknesses (10 mm and 4 mm) with multiple lengths and laminated materials.

2- Molding of Samples: The main method of preparation and casting of samples in the following stages:

• The weight of the amount of epoxy percentage required and hardener added by (2:1).

• The weight of the quantity of material reinforcement (pomegranate peel powder, eggshell powder, and palm frond powder) according to the indicated weight percentage.

• Mixing the material for reinforcement and matrix at room temperature. Where is mixing the weighted percentage of reinforcement and matrix in a special pot mixed by the electric mixer for a maximum of (1 – 10 minutes). However, this method does not give a good homogeneous mixture well, so notice the deposition of a large amount of material reinforcement at the bottom of the form after hardening.

• A new method is used to obtain a homogeneous mixture with very high specifications and very little precipitation was put mixing in Porcelain Mortar and Pestle. This method involves taking a part from the matrix (epoxy) and an estimated (20%) from the total weight fraction of epoxy only (without hardener) with the total reinforcement material and placed in Porcelain Mortar and Pestle we then process grinding and mixing at the same time and continue the process until we get on the mixture is semi-liquid and ensure the homogeneity of the combination at taking very simple part (diameter) by glass rod's head and put it on a glass slide for ensure homogeneity of the mixture and then put the amount remaining epoxy in Mortar and mix well with a glass rod and leave the mixture for an hour or two hours and up to 24 hours until smooth mixture then add the hardener and mix for two (2-3) minutes with note start warming mix.

• Pour the liquid combination into the center of the template to create a torrent that will flow to all parts of the ongoing and regular template, filling it to the desired level.

• Leave the mixing in the sample for 48 hours to harden completely, and then put it at oven temperature (50 C) for 5 hours to complete the formability.

3- Cut Up and Smoothing the Samples: Samples are cut to standard dimensions by the standards, and global measurements are displayed for every test in the table. Employing a band saw with soft teeth to guarantee that samples are cut without vibration and to prevent distortions from arising during the cutting process. Next comes the refinement step, where smooth sheets made of silicon carbide with varying degrees of softness are used.

3.3 Image Processing Approach

The image processing approach falls into the following stages (Fig. 3):

- FESEM image acquisition: Getting the high-resolution FESEM images with the size 3072*2188 for both treated and untreated images.
- Resizing FESEM images: FESEM images were resized into four sizes; D100 related to image size 3072*2188, D75 related to image size 2304*1641, D50 related to image size 1536*1094, and D25 related to image size 768*547.
- Texture surface measures: this step leads to extracting the image surface properties depending on the measuring of nine factors; contrast, dissimilarity, homogeneity, angular second moment, maximum probability, entropy, energy, mean, variance, and correlation.
- Decision Making: compare the obtained factors for all images to study the effect of the impact of hydrochloric acid on the samples.

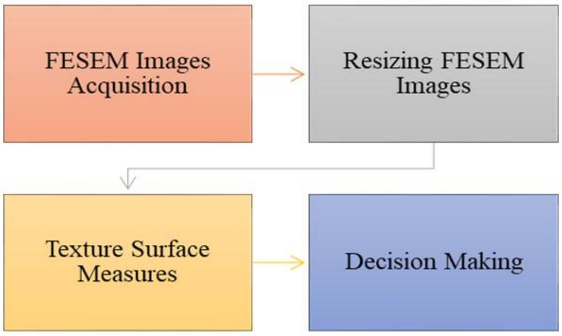


Figure 3. Image processing approach

4 Results and discussions

Nine factors were measured to demonstrate the effect of hydrochloric acid on the composite’s materials' surface structure. These factors are contrast, dissimilarity, homogeneity, angular second moment, maximum probability, entropy, energy, mean, variance, and correlation. These measures were applied to composite materials images obtained from field emission scanning electron microscopy (FESEM). Furthermore, these measures are applied before and after acid treatment. Four sizes of images were used in the comparison: D100, D75, D50, and D25. Different types of composite material images were used:

ES_Before: Egg Shells composite material before being treated with hydrochloric acid.

ES_After: Egg Shell composite material after treated with hydrochloric acid.

PF_Before: Palm Fronds composite material before treated with hydrochloric acid.

PF_After: Palm Fronds composite material after being treated with hydrochloric acid.

This section will focus on the obtained results for all factors applied to images with different sizes and for both egg shells composite material and palm frond composite material.

Image contrast refers to the variation in gray levels of an image. When there is a significant variation between the pixels values this leads to high contrast, on the other hand, low contrast indicates a minimal variation between the pixel values. Fig. 4 shows the distribution of image contrast measures for egg shells and palm frond composite materials before and after hydrochloric acid emersion. It is clear from the figure there is a slight increment of the image contrast in all cases of ES_After compared to ES_Before, but the increment is small in the case of PF_After compared to PF_Before. This leads to the Egg Shells composite materials are more affected by hydrochloric acid emersion compared with Palm Fronds composite materials.

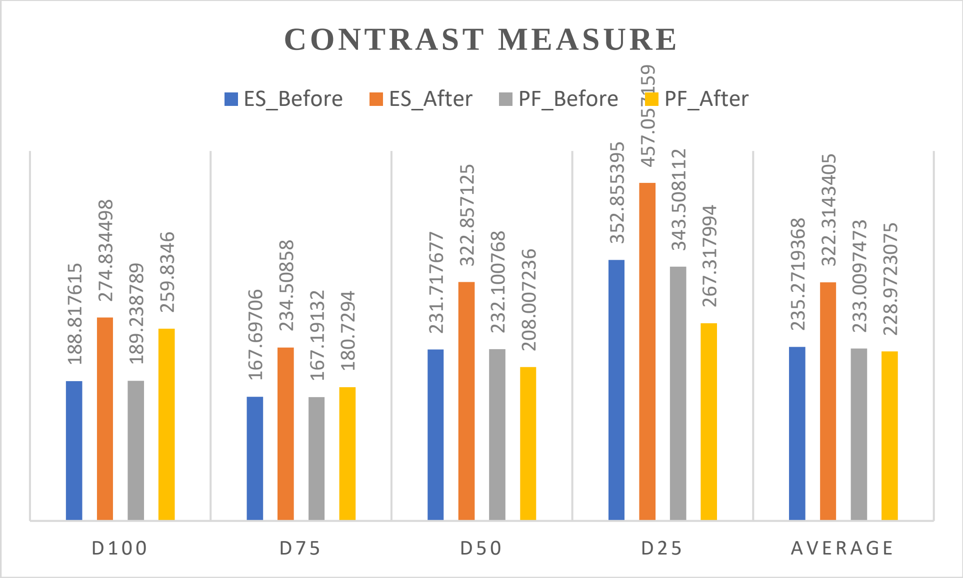


Figure 4. Contrast measure

The dissimilarity measure can be said to represent the difference between one-pixel values to the corresponding pixel value of an image. From this standpoint, it is possible to begin analyzing and explaining the results according to Fig. 5. From this figure, it can be observed that there is a significant increase in the dissimilarity factor in the egg shell sample after hydrochloric acid immersion. As for the palm frond sample, the value of the increase in the dissimilarity factor is less than the sample after hydrochloric acid immersion compared with the egg shell sample.

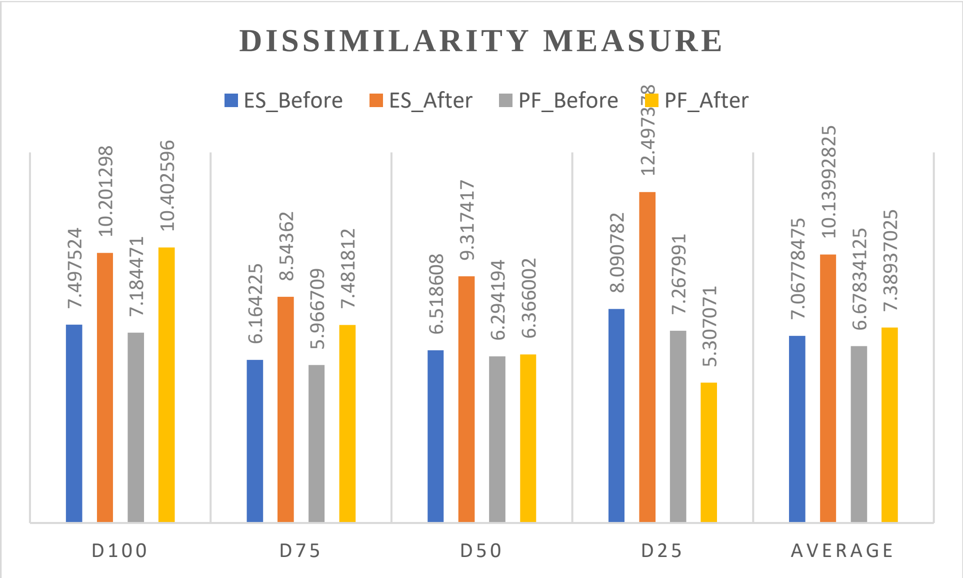


Figure 5. The dissimilarity measures

Homogeneity measure is an important measure of image texture that refers to the uniformity of pixel distribution within an image. It indicates the degree to which the pixels in the image are similar to each other. When it has a value near 1 it indicates high uniformity, while it is valued closer to 0 indicating low uniformity. Fig. 6 shows the change in the homogeneity value before and after hydrochloric acid immersion for the egg shell and palm frond samples. This figure shows that the egg shell sample is little affected by hydrochloric acid immersion compared to the palm frond sample, which is greatly affected by hydrochloric acid immersion.

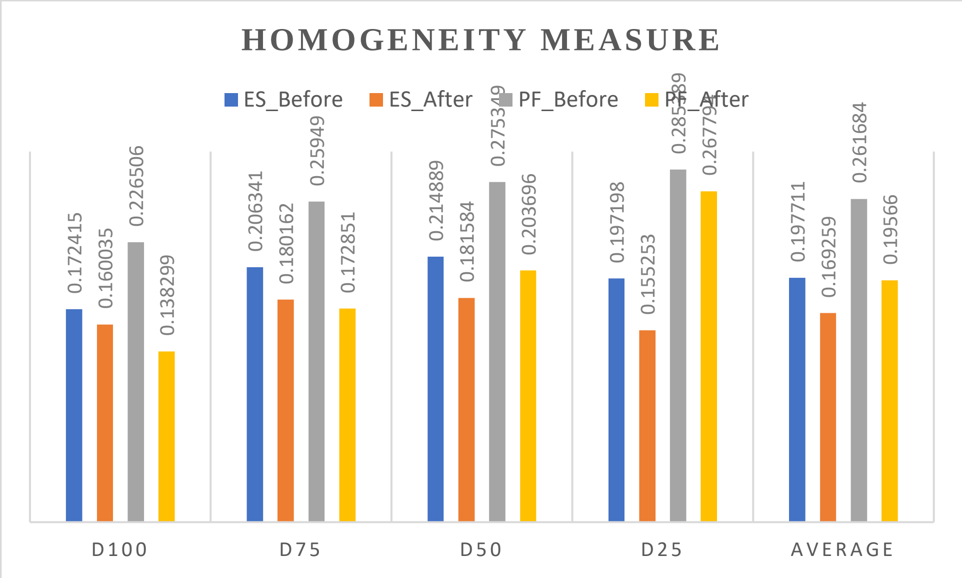


Figure 6. Homogeneity measure

Angular second-moment value evaluates the distribution of pixel values in various directions within the image. The obtained values of Angular second moment for both cases (egg shell and palm frond) are small indicating a lack of pixel distribution in various directions as demonstrated in Fig. 7. This figure references that the egg shell sample is less affected by hydrochloric acid immersion than the palm frond sample, which is greatly affected by hydrochloric acid immersion. Through this measurement, it is clear that the distribution of pixel directions is greatly affected after hydrochloric acid immersion of the palm frond sample.

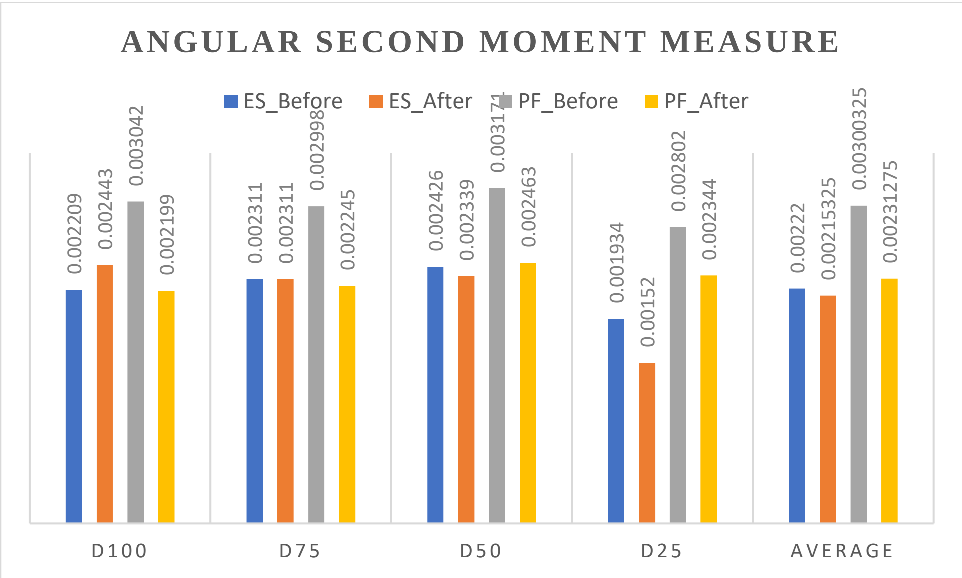


Figure 7. Angular second moment measure

Maximum probability refers to the most frequent pixel intensity in the image. Fig. 8 shows that the value of the maximum probability does not change in the egg shell sample after hydrochloric acid immersion. This leads us to the stability of the egg shell sample against hydrochloric acid, while there is a noticeable change in the maximum probability values for the palm frond sample after hydrochloric acid immersion.

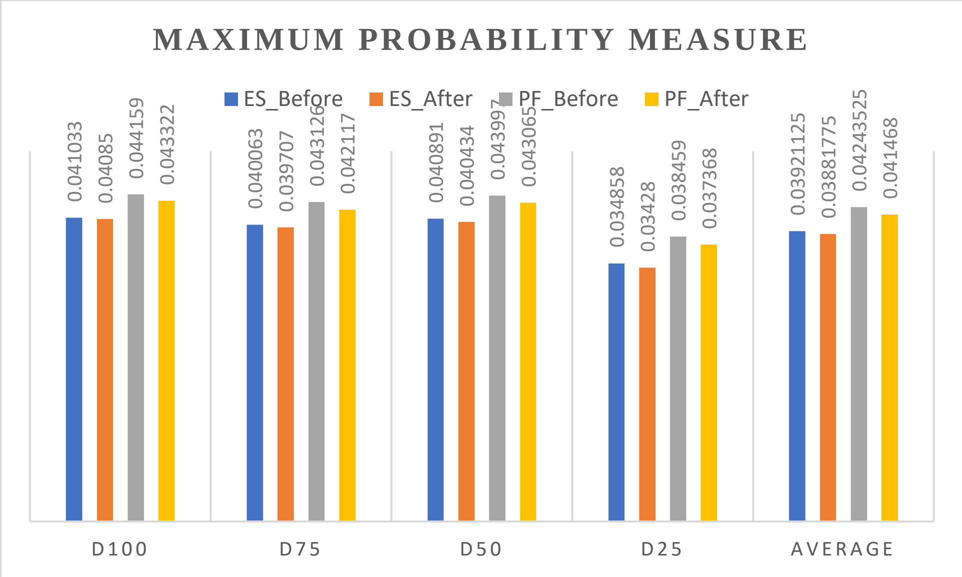


Figure 8. Maximum probability measure

Entropy is a measure of pixel intensity randomness in the distribution within the image. The high value of entropy indicates high complexity in the image, while the low value of entropy indicates greater low complexity. In general, Fig. 9 references that the entropy values in the two samples are of mid values. This figure shows the effect of acid immersion on the palm frond sample is less than its effect on the egg shell sample, meaning that the palm frond sample is less complex after acid immersion.

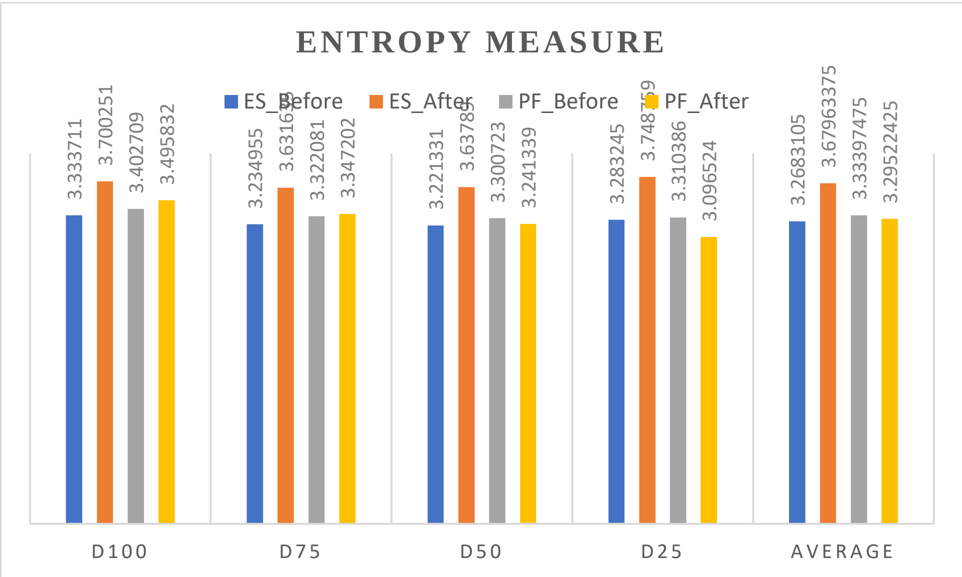


Figure 9. Entropy measure

Energy sometimes indicates to angular second moment, also it refers to visual features intensity pixels in an image. As a definition, when returning to the angular second-moment measurements, it is possible to notice a similarity in the change occurring in the examined samples in Fig. 7 and 10. Fig. 10 shows that the energy in the palm frond sample decreases significantly after acid immersion, but not by much, and that the egg shell sample is very little affected by acid immersion.

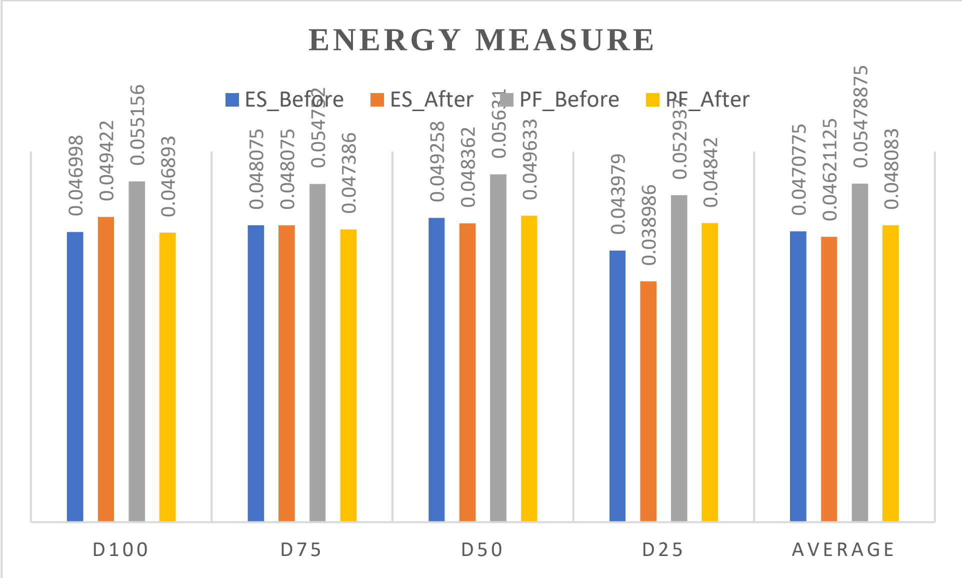


Figure 10. Energy measures

Image mean value represents the sum of intensity pixel values divided by the total number of pixels. Fig. 11 demonstrates that the mean value is clearly and significantly affected in the case of the egg shell sample after acid immersion and that its value increases as a result. In the case of the palm frond sample, its effect on acid immersion is slight, meaning that it resists the acidic environment.

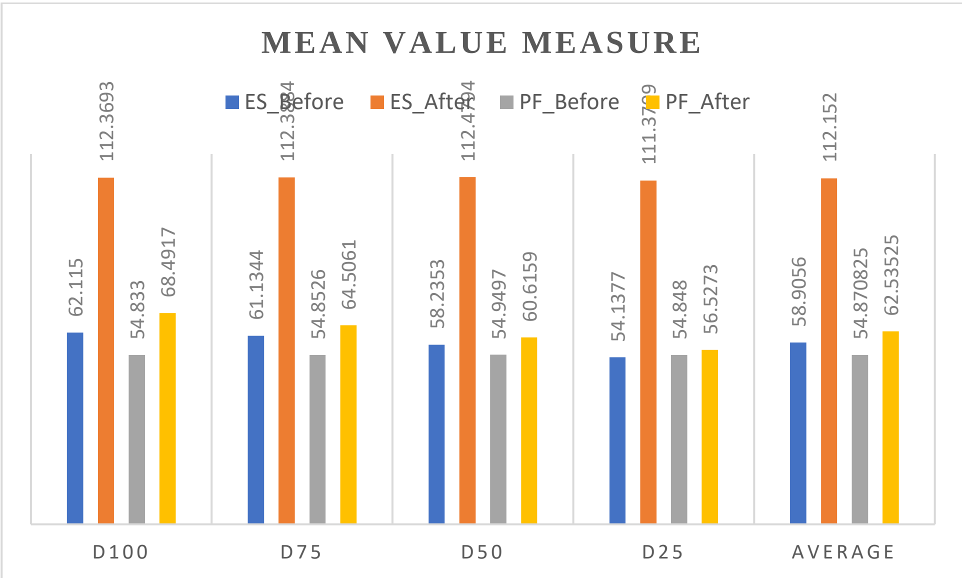


Figure 11. Mean value measure

Variance refers to the distribution of pixel intensity within an image. It also indicates the deviation of pixel values from the average intensity value. Referring to Fig. 12, it can be said that the two samples of egg shells and Palm fronds are affected by acid immersion, and this is evident by observing the variance factor for the two samples before and after acid immersion. In addition, we note that the effect of the egg shell sample is greater and reaches 73%, while the effect of the palm frond sample reaches 62%.

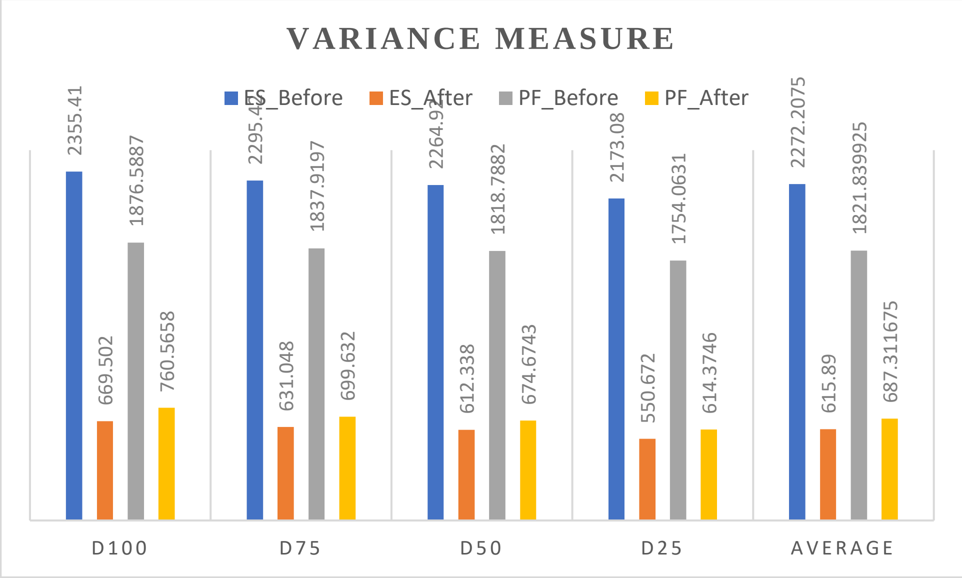


Figure 12. Variance measure

Correlation denotes a measure of the linear dependence of the gray levels of neighboring pixels. The correlation value for the egg shell and Palm fronds samples is characterized by a high correlation factor as displayed in Fig. 13. They are slightly affected by acid immersion, but the egg shell sample shows a slight increase in the correlation factor, which references the consistency of the sample.

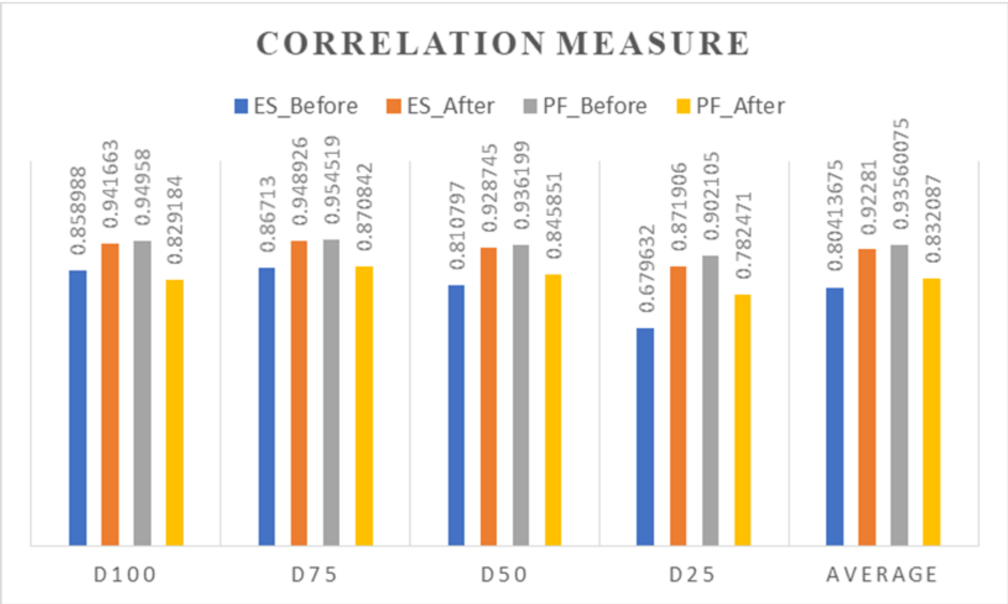


Figure 13. Correlation measure

5 Conclusion

Introducing image processing operations into surface analysis of composite materials helps to recognize the features of the surfaces of these materials and discover the extent of the cohesion of the samples. The field emission scanning electron microscopy used to generate digital images has given the work strength and accuracy. These images associated with egg shell and palm frond samples were analysed before and after acid immersion. Several features are calculated from a grayscale image that helps us understand the image in general. Multiple statistical measurements were performed on the samples to determine their effect on the acid solution. Through these measurements, it was found that the egg shell and palm frond samples have resistance to the acid solution, and rather than the resistance of the palm frond sample was significantly greater than the resistance of the egg shell sample. Advanced image analysis to extract quantitative data from FESEM images could be employed in future work combined with nanotechnology.

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