

Automated Scanning Electron Microscopy Image Analysis of Molybdenum–Iron Oxide Nanocomposites for Structure–Property Characterization Using Cellpose Segmentation and Machine Learning Classification

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Abstract

Structural properties of metal oxide nanocomposites play a crucial role in their performance in catalytic, environmental, and energy-related applications. This study presents an automated framework for the quantitative analysis of morphological and texture features of molybdenum-iron oxide nanocomposites using scanning electron microscopy (SEM) images. Instance segmentation was performed using the Cellpose deep learning model based on a U-Net architecture, enabling accurate separation of individual nanocomposites from dense and complex microstructures. Post-segmentation, 10 quantitative features, comprising six morphological descriptors (equivalent diameter, perimeter, aspect ratio, circularity, solidity, and eccentricity) and four Gray-Level Co-occurrence Matrix texture descriptors (contrast, homogeneity, energy, and correlation), were extracted. The resulting feature dataset was used to train four supervised machine learning classifiers: Random Forest, Support Vector Machine, eXtreme Gradient Boosting, and Light Gradient-Boosting Machine. Model performance was evaluated using accuracy, precision, recall, and F1-score metrics. The gradient boosting models demonstrated superior classification performance, with Light Gradient-Boosting Machine achieving the highest mean accuracy of 95.1% for shape classification, while eXtreme Gradient Boosting achieved the highest mean accuracies of 96.9% for size range classification and 94.8% for texture classification. These results highlight the effectiveness of gradient boosting techniques in capturing complex relationships between morphological and texture features for automated nanocomposite characterization. Overall, the proposed framework enables efficient automated molybdenum-iron oxide SEM image analysis and quantitative nanocomposite characterization, providing deeper insight into structure-property relationships in advanced nanomaterials.

Categories: Image Processing and Analysis, Machine Learning (ML), Deep Learning

Keywords: molybdenum–iron oxide nanocomposites, sem, cellpose, u-net, instance segmentation, morphology, texture analysis, glcm, machine learning, lightgbm

Introduction

It is evident that nanocomposite materials with transition metal oxides have attracted significant attention due to their catalytic, magnetic, and electrochemical properties. The molybdenum-iron oxide nanocomposites among these materials have emerged as a good prospect since they combine the redox and magnetic properties of iron oxides with the catalytic

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properties of molybdenum oxides. The size, shape, and surface morphology of nanocomposites of such nanomaterials have a decisive effect on their performance, as they are directly related to surface area, active sites, and interfacial interactions. Scanning electron microscopy (SEM) is also an extensive technique for studying the morphology of nanomaterials due to the high-resolution images it offers, which can provide insight into particle size, size distribution, shape variation, and agglomeration. Traditional SEM analysis depends largely on manual evaluation, making it time-consuming, subjective, and prone to human bias. Large datasets also cannot be measured manually, which restricts the scalability and reproducibility of nanocomposite characterization. Recent developments in artificial intelligence have made it possible to perform automated image analysis, which can be used to obtain quantitative data from microscopic images. Deep learning techniques, such as the U-Net architecture, have demonstrated impressive performance in the segmentation of particles from complex nanostructures using SEM/transmission electron microscopy images. In addition to segmentation, the quantitative characterization of nanocomposites requires the derivation of descriptive and meaningful features that describe geometrical, morphological, and texture characteristics of nanocomposites. The area, perimeter, equivalent diameter, circularity, eccentricity, and texture features based on the Gray-Level Co-occurrence Matrix (GLCM) are also useful for gaining an understanding of the morphology and surface structure of nanocomposites. These features can be evaluated using machine learning algorithms to classify nanomaterials and analyze them without human intervention. There is an increased application of machine learning methods in predicting, classifying, and optimizing nanomaterials. Ensemble-based and gradient boosting algorithms have exhibited a high level of effectiveness in processing complex and high-dimensional data, making them suitable for nanomaterial characterization. Even with these developments, there are no comprehensive models uniting deep learning-based segmentation, feature extraction, and machine learning classification of molybdenum-iron oxide nanocomposites through automated SEM analysis. Thus, the proposed study will offer a fully automated pipeline that incorporates Cellpose-based instance segmentation and machine learning classification of molybdenum-iron oxide nanocomposites using SEM images. The suggested framework will offer a robust and scalable approach to understanding morphology-inspired structure-property relationships in advanced nanomaterials.

Molybdenum oxides have received considerable interest because of their multiple oxidation states, layered structure, and tunable electronic properties. Bhattacharjee and Roy [1] reviewed the chemistry of molybdenum oxides and highlighted that catalytic and electrochemical activity can be tailored through polymorphic phases and controlled oxygen vacancy engineering. Shaikh et al. [2] discussed the oxidation states of molybdenum and emphasized their role in designing hybrid and doped oxide nanostructures. Kamoun et al. [3] synthesized molybdenum oxide-silica nanocomposites using chemical methods and showed that compositional tuning enhances surface area and photoactivity. Nanostructured molybdenum oxides derived from intermetallic compounds were reported by Raj et al. [4], demonstrating high efficiency for hydrogen evolution reactions. Shujah et al. [5] synthesized molybdenum-doped iron oxide nanostructures via co-precipitation and reported enhanced dye degradation and antimicrobial activity. Lima et al. [6] synthesized Fe-doped α - MoO_3 and observed induced magnetic behavior due to structural modification. Hybrid molybdenum-iron oxide materials have also been widely explored for electrochemical applications. Shit et al. [7] developed S-doped FeMoO_4 , showing enhanced electrocatalytic water splitting due to improved electron transfer and increased active sites. Chen and Qin [8] investigated MoO_x - Fe_2O_3 /graphene composites as lithium-ion battery anodes, while Imran et al. [9] demonstrated improved conductivity and catalytic activity in mixed transition-metal oxides. Concepción and de Melo [10] summarized synthesis routes and applications of molybdenum oxides, highlighting the importance of heterostructures and doping. Morphology and crystal phase strongly influence catalytic performance. Zhang et al. [11] showed morphology-dependent catalytic behavior of Fe_2O_3 nanostructures, while Tasnim et al. [12] emphasized morphology-controlled antimicrobial performance of metal-doped iron oxide nanoparticles. Sawant et al. [13] identified transition-metal molybdates as promising supercapacitor materials using machine learning analysis, and Robertson et al. [14] highlighted ML-assisted design of MoSe_2 nanostructures for energy storage.

Overall, particle size, morphology, phase composition, and surface texture strongly influence photocatalytic, antimicrobial, electrocatalytic, and energy-storage performance. Microscopic image analysis has become a major research area for nanomaterial characterization. Lopez Gutierrez et al. [15] applied neural networks for nanoparticle detection in SEM images. Bals and Epple [16] used deep learning for nanoparticle size and shape analysis, and Zelenka et

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al. [17] demonstrated automated nanoparticle classification using deep learning. Shah et al. [18] applied U-Net networks for graphene SEM segmentation, while Sowmya Sanan and Rimal Isaac [19] proposed a transfer learning-based U-Net for nanoparticle boundary detection. Bai et al. [20] introduced semantic segmentation for agglomerate detection in nanocomposites. Mohsenzadeh et al. [21] used deep learning for morphological particle segmentation and interfacial property prediction. Cellpose-based approaches were demonstrated by Riendeau et al. [22] and Merouane et al. [23] for reliable microscopy segmentation and particle size measurement. Machine learning has also been widely adopted for nanoparticle classification and property prediction. Ju et al. [24] developed frameworks for oxide nanomaterial classification. Bannigidad et al. [25] demonstrated metal oxide nanoparticle and nanocomposite SEM image analysis and classification using various machine learning algorithms. Tymoshchuk et al. [26] compared several supervised machine learning algorithms for composite material classification. Their results showed that neural network-based models provided more accurate and reliable predictions than conventional classifiers. Buckman and Gundlach-Graham [27] proposed a two-stage semi-supervised machine learning framework for nanoparticle classification. Their approach effectively reduced classification errors caused by noisy data and improved the reliability of nanoparticle identification. Jahanian et al. [28] demonstrated that machine learning can effectively classify nanomaterials based on their morphological characteristics. The study highlights the potential of AI-assisted image analysis for rapid and reliable nanomaterial identification. Robertson and Lopez [29] developed a machine learning framework for classifying nanoparticles based on their morphological features, demonstrating reliable shape recognition with reduced manual intervention. Sushma et al. [30] compared machine learning techniques for nanoparticle morphology characterization. Banerjee et al. [31] used machine learning to predict nanoparticle transport behavior, and Khodadadi et al. [32] applied ensemble machine learning algorithms for nanoparticle property prediction. Liu et al. [33] demonstrated machine learning-assisted synthesis of iron oxide particles. Rawat et al. [34] reviewed machine learning applications in rare-earth-doped oxide nanoparticles. Ma et al. [35] applied interpretable machine learning to predict antitumor effects of metal oxide nanoparticles, while Mary and Mujeeb [36] predicted antibacterial activity using machine learning. Vargo et al. [37] and Bannigidad et al. [38] proposed a machine learning-based digital image analysis framework for quantitative morphological analysis of metal oxide nanoparticles and nanocomposites. Barnard and Opletal [39] compared multiple feature sets and machine learning algorithms to identify optimal models for metallic nanoparticle analysis, demonstrating that feature selection plays a crucial role in prediction accuracy. Ju et al. [24] evaluated several supervised machine learning algorithms to predict the behavior of oxide nanomaterials. The study identified ensemble-based models as the most effective approach for achieving robust and accurate classification. These studies collectively demonstrate the growing role of machine learning in nanomaterial design, synthesis, characterization, classification, and performance prediction.

Although there is extensive research on molybdenum-iron oxide nanocomposites, the majority of studies are mainly focused on synthesis and functional performance, while quantitative morphological characterization of nanocomposites based on SEM images is still largely manual and time-consuming. Several deep learning methods are based on hand-labeled datasets, which restrict scalability and reproducibility. Moreover, texture-based characterization is frequently not given enough attention, and little research offers a comparative analysis of various machine learning algorithms on a common dataset. Currently, annotation-free segmentation, quantitative feature extraction, and multi-task machine learning classification of molybdenum-iron oxide nanocomposites using SEM images have not been developed. This paper presents a fully automated system to analyze molybdenum-iron oxide nanocomposites using SEM images based on their morphology and texture characteristics.

Materials And Methods

Materials

Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), ferrous sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$), sodium hydroxide (NaOH), and ethanol were purchased from commercial suppliers and used without further purification. Deionized water was used throughout the synthesis process.

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Preparation of molybdenum-iron oxide nanocomposites

Molybdenum-iron oxide nanocomposites were synthesized using a co-precipitation method, which is widely used for preparing mixed metal oxide nanomaterials. Initially, an aqueous solution containing ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was prepared in deionized water under continuous magnetic stirring. The molar ratio of Fe^{3+} to Fe^{2+} was maintained at 2:1 to promote the formation of magnetite (Fe_3O_4). Sodium molybdate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) was then added to the solution as the molybdenum precursor. The reaction mixture was heated to 70–80°C, and a 1 M NaOH solution was slowly added dropwise under vigorous stirring until the pH reached approximately 10–11, leading to the formation of a dark precipitate. The mixture was maintained at this temperature for 2 hours to allow complete nucleation and growth of the nanocomposites. The resulting precipitate was separated by centrifugation and washed several times with deionized water and ethanol to remove residual ions and impurities. The product was then dried in an oven at 80°C for 12 hours to obtain the final molybdenum-iron oxide nanocomposite powder.

SEM characterization

The surface morphology and microstructure of the synthesized molybdenum-iron oxide nanocomposites were examined using SEM. Prior to imaging, a small amount of the nanocomposite powder was dispersed in ethanol using ultrasonication and drop-cast onto a conductive substrate. The samples were dried and coated with a thin conductive layer to minimize charging effects during imaging. SEM images were recorded at different magnifications to capture nanoscale morphological details of the nanocomposite particles. These images were used as input data for the automated analysis framework developed in this study. The collected SEM micrographs were further processed for image preprocessing, segmentation, feature extraction, and machine learning analysis.

Dataset overview

The dataset consists of SEM images of molybdenum-iron oxide nanocomposites used for automated nanocomposite analysis and classification. Nanocomposite segmentation was performed using a U-Net-based Cellpose deep learning model to accurately isolate individual nanocomposite particles from the SEM micrographs. A total of 324 nanocomposite particles were successfully segmented and analyzed. From each segmented particle, 10 quantitative features were extracted to describe morphological and surface characteristics. These features were grouped into three major categories: size, shape, and texture. Size-related descriptors include area and equivalent diameter, while shape descriptors include perimeter, aspect ratio, eccentricity, and circularity. Surface characteristics were captured using GLCM texture features such as contrast, homogeneity, energy, and correlation. This structured dataset was used to train and evaluate machine learning models for automated classification of nanocomposite particles based on shape, size class, and surface texture features. Table 1 summarizes the molybdenum-iron oxide SEM image dataset.

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Parameter	Description
Material	Molybdenum-Iron Oxide Nanocomposites
Imaging technique	SEM
Segmentation method	U-Net-based Cellpose Model
Total nanocomposite particles analyzed	324
Total extracted features	10
Feature categories	Size, Shape, Texture

TABLE 1: Molybdenum-iron oxide nanocomposite SEM dataset description
SEM, Scanning Electron Microscopy

Proposed method

The proposed method aims to automate the analysis of molybdenum-iron oxide nanocomposites from SEM images by integrating deep learning-based segmentation with feature extraction and machine learning classification. Initially, SEM images are preprocessed to improve image quality and remove noise. A deep learning segmentation model is then applied to accurately identify and segment nanocomposites from the SEM images. Following segmentation, morphological and texture features such as particle size, shape descriptors, and texture parameters are extracted from the segmented particles. These extracted features are subsequently used as inputs for machine learning algorithms to classify and analyze the nanocomposite structures. This integrated approach enables efficient, objective, and reproducible analysis compared to traditional manual SEM evaluation. Figure 1 presents the complete workflow for the analysis of the molybdenum-iron oxide nanocomposites. All these steps are summarized in the following algorithm.

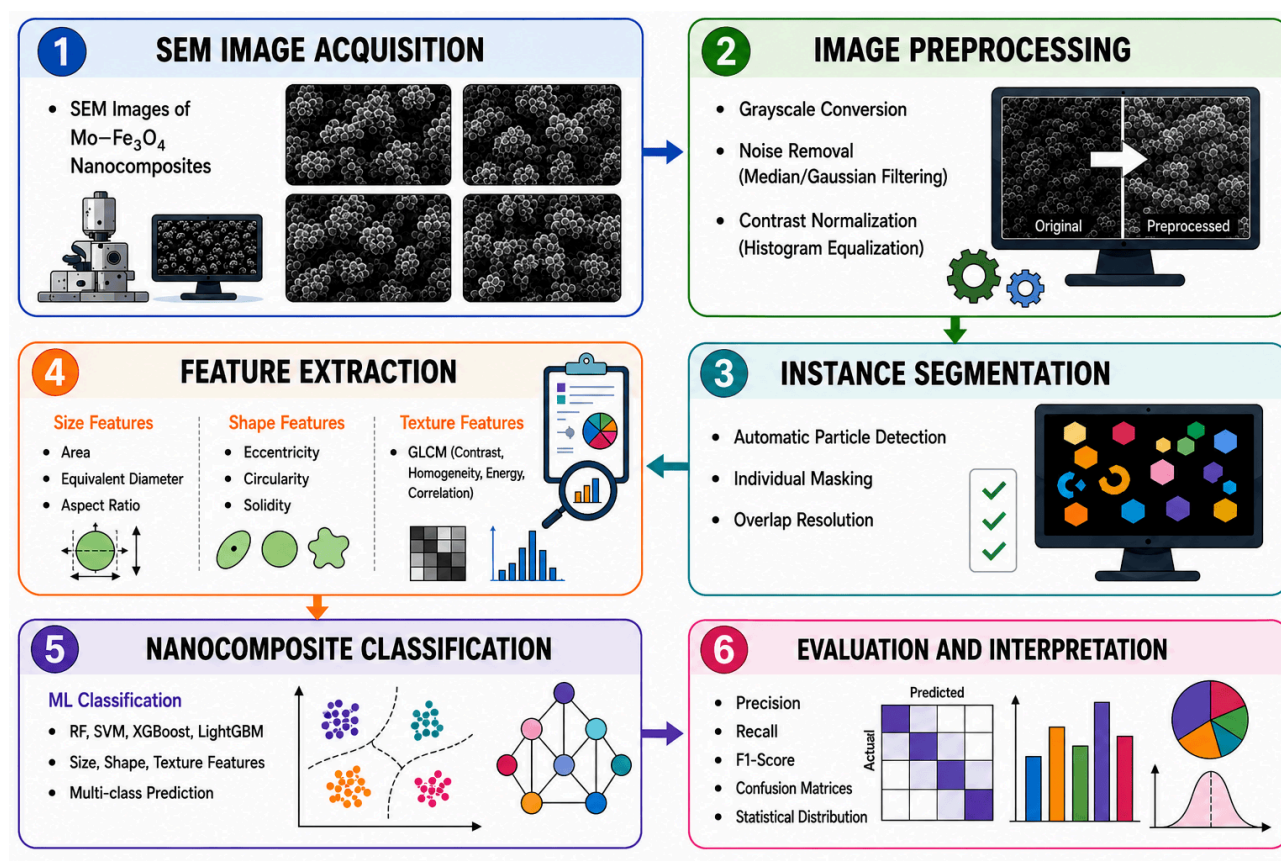


FIGURE 1: Workflow diagram of molybdenum-iron oxide nanocomposites analysis

GLCM, Gray Level Co-occurrence Matrix; ML, Machine Learning; RF, Random Forest; SEM, Scanning Electron Microscopy; SVM, Support Vector Machine

Algorithm for molybdenum-iron oxide nanocomposite analysis

Step 1: Load the molybdenum-iron oxide SEM image and convert it to grayscale.

Step 2: Remove noise and apply CLAHE (Contrast-Limited Adaptive Histogram Equalization) to enhance contrast.

Step 3: Convert the image to RGB format for compatibility with the Cellpose model input.

Step 4: Generate segmentation masks for individual nanocomposites using the Cellpose U-Net model.

Step 5: Combine masks and generate a colored overlay for visual verification of nanocomposite isolation.

Step 6: Label segmented nanocomposites and extract quantitative features: area, perimeter, aspect ratio, eccentricity, circularity, equivalent diameter (for shape/size), and GLCM texture features, including contrast, homogeneity, energy, and correlation.

Step 7: Construct a feature matrix and assign shape, size class, and surface texture labels.

Step 8: Split the dataset into training (80%) and testing (20%) sets.

Step 9: Train the specified machine learning models: Random Forest, XGBoost, Support Vector Machine (SVM) and LightGBM for shape, size class, and surface texture classification.

Step 10: Evaluate model performance using F1-score, recall, precision, and accuracy.

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Preprocessing of molybdenum-iron oxide nanocomposite SEM images

SEM images of molybdenum-iron oxide nanocomposites were preprocessed before segmentation to increase the visibility of nanocomposites and reduce noise in the images. The preprocessing involved conversion to grayscale, contrast enhancement using histogram equalization, noise suppression using a Gaussian filter, and intensity normalization. These operations enhanced edge visibility and improved efficient boundary identification during the segmentation process.

Cellpose-based instance segmentation

A pretrained Cellpose model based on a U-Net-like encoder-decoder architecture was employed for segmenting nanocomposites from SEM images, as illustrated in Figure 2. The input SEM image is processed through the encoder, consisting of convolutional layers (3×3), ReLU activation, batch normalization, and max-pooling for feature extraction. The bottleneck layer captures high-level features, which are then passed to the decoder for spatial reconstruction using up-convolution (2×2) and convolutional layers. Skip connections between corresponding encoder and decoder layers help preserve fine structural details. The network generates output maps that enable instance-level segmentation, where each nanocomposite structure is uniquely identified, allowing accurate mask generation. A set of instance masks was created using these outputs, enabling the separation of overlapping and closely packed nanocomposites.

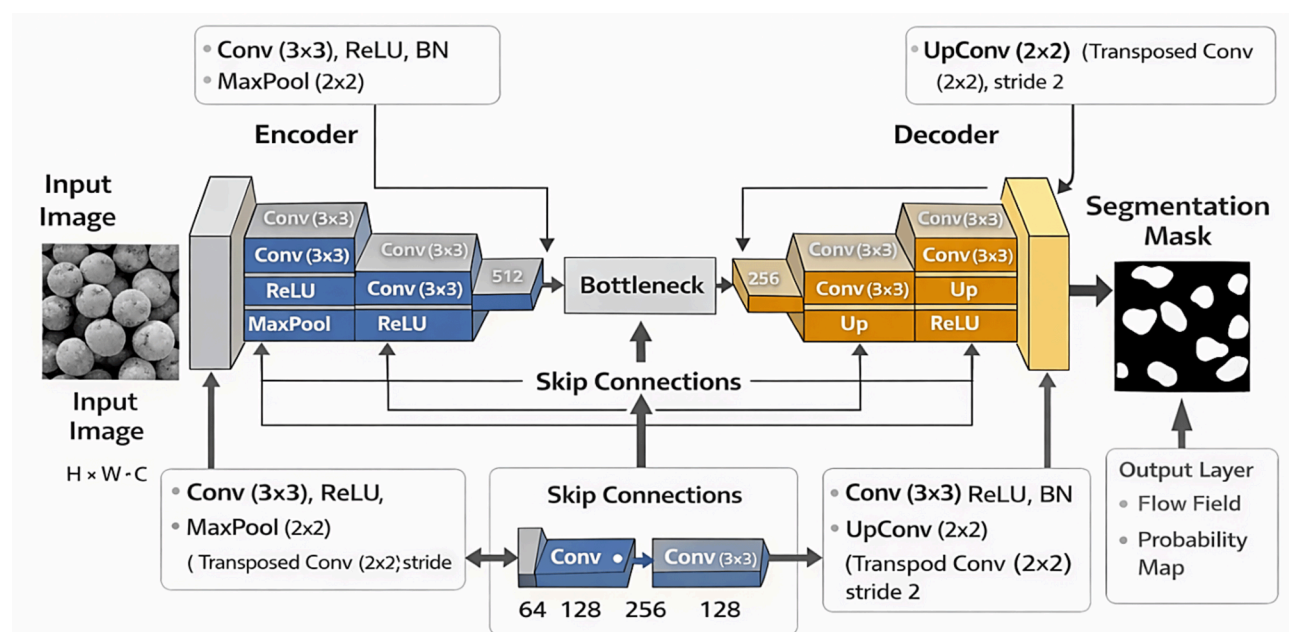


FIGURE 2: U-Net-based cellpose model architecture

Feature extraction

The quantitative features of nanocomposites, such as size, shape, and surface texture, were obtained from each segmented nanocomposite. Table 2 presents the morphological and texture features extracted from the segmented nanocomposites. Nanocomposite size was expressed in terms of equivalent circular diameter, which is the diameter of a circle with an area equivalent to that of the segmented nanocomposite particle. Circularity was used to measure shape compactness and its similarity to a perfect circle. Aspect ratio, which is the ratio of the actual length of the major axis to the minor axis of the fitted ellipse, was used to define particle elongation. Along with these main features, other morphological characteristics, such as solidity and eccentricity, were calculated using common techniques of region-based image analysis to obtain boundary regularity and non-circular geometry. The GLCM was used to obtain texture features based on the statistical texture model suggested by Haralick et al. [40]. To quantify surface roughness and

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contrast, as well as the energy and correlation of surfaces, texture descriptors such as contrast, homogeneity, energy, and correlation were computed for each nanocomposite region. GLCM feature values were grouped into three texture classes using quantile-based thresholding. The 33rd and 66th percentile values of GLCM contrast and GLCM homogeneity served as the threshold values. Samples with high contrast and low homogeneity were labeled as Rough, those with low contrast and high homogeneity were labeled as Smooth, and the remaining samples were classified as Moderately Rough. All the obtained features were gathered into a structured dataset, where each row is associated with a single nanocomposite and each column represents a quantitative characteristic.

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Particle ID	Size (nm)	Perimeter (nm)	Aspect Ratio	Circularity	Solidity	Eccentricity	Contrast	Homogeneity	Energy	Correlation
1	46.09	182.18	2.48	0.63	0.92	0.92	2856.756	0.309	0.299	0.715
2	54.04	193.39	1.97	0.77	0.95	0.86	1853.538	0.26	0.248	0.634
3	64.45	224.25	1.18	0.82	0.93	0.53	2037.789	0.205	0.19	0.637
4	37.85	125.96	1.43	0.89	0.96	0.72	2351.054	0.265	0.249	0.714
5	97.44	347.89	1.27	0.77	0.96	0.61	2880.824	0.329	0.274	0.843
6	101	444.96	3.37	0.51	0.96	0.96	2651.859	0.286	0.271	0.756
7	26.16	95.36	2.29	0.74	0.9	0.9	3419.225	0.267	0.264	0.581
8	113.05	482.38	1.52	0.54	0.83	0.75	1829.171	0.512	0.471	0.887
9	60.04	225.71	2.41	0.7	0.96	0.91	1629.017	0.366	0.342	0.84
10	95.37	349.76	1.64	0.73	0.93	0.79	2089.83	0.218	0.198	0.777
11	77.05	269.53	1.03	0.81	0.87	0.26	2177.173	0.443	0.414	0.872
-	-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-	-	-	-
321	65.73	263.48	1.95	0.61	0.89	0.86	1605.74	0.287	0.262	0.757
322	68.46	239.25	1.7	0.81	0.98	0.81	910.793	0.366	0.342	0.612
323	48.86	175.71	1.3	0.76	0.92	0.64	1348.483	0.304	0.287	0.605

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324	49.02	187	1.44	0.68	0.9	0.72	793.012	0.429	0.413	0.431
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TABLE 2: Morphological and texture features of molybdenum-iron oxide nanocomposites

The particle size was represented using the equivalent diameter, which corresponds to the diameter of a circle having the same area as the particle. The equivalent diameter is given by Eq. (1):

$$\text{Equivalent Diameter} = \sqrt{\frac{4A}{\pi}} \quad (1)$$

The morphology of the particles was characterized using multiple shape descriptors that capture geometric properties such as compactness, elongation, and boundary irregularity.

The circularity, which measures the closeness of a particle to a perfect circle, is given by Eq. (2):

$$\text{Circularity} = \frac{4\pi A}{P^2} \quad (2)$$

where P represents the perimeter of the nanocomposite particle. Eq. (2) indicates that values closer to unity correspond to highly circular particles.

The aspect ratio, which describes the elongation of the nanocomposite particle, is given by Eq. (3):

$$\text{Aspect Ratio} = \frac{L_{major}}{L_{minor}} \quad (3)$$

where L_{major} and L_{minor} denote the major and minor axis lengths, respectively. Eq. (3) helps distinguish elongated particles from symmetric ones.

The solidity, which reflects the compactness of the particle, is given by Eq. (4):

$$\text{Solidity} = \frac{A}{A_{convex}} \quad (4)$$

where A_{convex} is the area of the convex hull. Eq. (4) indicates the degree of irregularity in particle boundaries.

The eccentricity, which measures the deviation of the particle from a circular shape, is given by Eq. (5):

$$\text{Eccentricity} = \sqrt{1 - \frac{b^2}{a^2}} \quad (5)$$

where a and b represent the semi-major and semi-minor axes, respectively. Eq. (5) shows that values closer to 1 correspond to highly elongated particles.

Texture features were extracted using the GLCM based on Haralick descriptors to capture spatial intensity variations within the particles. The contrast, which measures local intensity variations, is given by Eq. (6):

$$\text{Contrast} = \sum_{i,j} (i - j)^2 P(i, j) \quad (6)$$

where $P_{i,j}$ denotes the normalized GLCM value. Eq. (6) highlights the degree of intensity variation in the image.

The homogeneity, which measures the similarity of pixel intensities, is given by Eq. (7):

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$$\text{Homogeneity} = \sum_{i,j} \frac{P(i,j)}{1 + |i - j|} \quad (7)$$

Eq. (7) indicates that higher values correspond to more uniform textures.

The energy, which represents the uniformity of the texture distribution, is given by Eq. (8):

$$\text{Energy} = \sum_{i,j} P(i,j)^2 \quad (8)$$

Eq. (8) measures the consistency of gray-level distribution within the particle.

The correlation feature measures the linear dependency of gray-level values between neighboring pixels in the GLCM. It is computed using Eq. (9), as shown below:

$$\text{Correlation} = \sum_i \sum_j \frac{(i - \mu_x)(j - \mu_y) P(i,j)}{\sigma_x \sigma_y} \quad (9)$$

Higher correlation values indicate a stronger linear relationship between neighboring pixel intensities, whereas lower values correspond to weaker spatial dependencies.

Results And Discussion

Preprocessing and segmentation results

Representative SEM images of the molybdenum-iron oxide nanocomposites were preprocessed using grayscale conversion, Gaussian filtering, and histogram equalization to enhance image quality. These preprocessing steps improved contrast, reduced background noise, and clearly distinguished individual structures within the nanocomposites. The preprocessed images were subsequently analyzed using the Cellpose model for instance segmentation. The model accurately identified and segmented 324 individual particles, including overlapping regions, generating precise segmentation masks for quantitative morphological and texture feature extraction. The complete process of image preprocessing and nanocomposite segmentation is shown in Figure 3.

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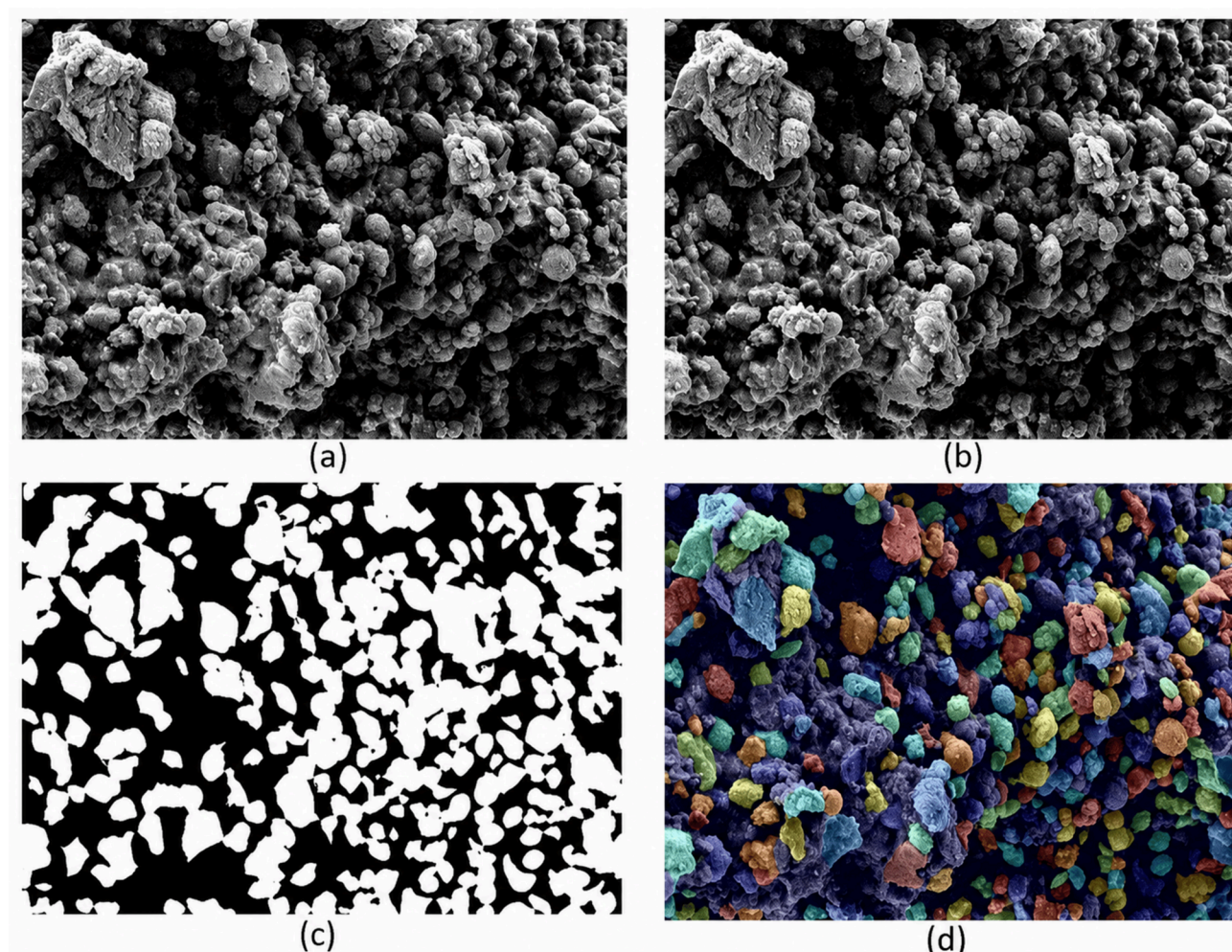


FIGURE 3: Sample SEM image of molybdenum-iron oxide nanocomposites. a) Original image. b) Preprocessed image. c) Binary mask image. d) Segmented image

SEM, Scanning Electron Microscopy

Size, shape, and texture distribution of nanocomposite particles

The proportion of nanocomposite particles of different sizes and shapes is given in Table 3. Most of the nanocomposite particles were found to be in the 51–100 nm (65.12%), 101–150 nm (19.44%), 0–50 nm (12.04%), and 151–200 nm (3.40%) size ranges. The above distribution implies that the synthesis process yielded mostly particles within the desirable nanoscale range of less than 150 nm. The low proportion of larger particles indicates controlled growth kinetics and slight agglomeration during synthesis. Morphological examination has shown that a significant percentage of nanocomposite particles exhibited an elliptical morphology (66.67%), followed by spherical and polygonal particles at 29.94% and 3.40%, respectively. The dominant occurrence of elliptical morphologies implies anisotropic crystal growth behavior, which is usually observed in transition metal oxide nanocomposites. Based on the extracted GLCM texture features, the nanocomposite particles were further categorized into three texture classes: smooth (33.02%), moderately rough (33.95%), and rough (33.02%). The texture classes were nearly equally distributed, indicating a balanced representation of different surface characteristics in the dataset.

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Classification Type	Class	Number of Nanocomposites	Percentage (%)
Size	0–50 nm	39	12.04
	51–100 nm	211	65.12
	101–150 nm	63	19.44
	151–200 nm	11	3.40
Shape	Elliptical	216	66.67
	Spherical	97	29.94
	Polygonal	11	3.40
Texture	Smooth	107	33.02
	Moderately Rough	110	33.95
	Rough	107	33.02

TABLE 3: Molybdenum-iron oxide nanocomposites distribution by size, shape, and texture

Shape classification performance

The performance of Random Forest, SVM, XGBoost, and LightGBM for molybdenum-iron oxide nanocomposite shape classification was evaluated using five-fold cross-validation, and the quantitative results are summarized in Table 4. As shown in Table 4, LightGBM achieved the highest mean accuracy (95.1%), followed by XGBoost (94.7%), Random Forest (94.3%), and SVM (93.6%). LightGBM also recorded the highest precision (0.922), recall (0.951), and F1-score (0.936), with a low standard deviation (0.004) and a narrow 95% confidence interval (0.946-0.956), indicating stable and reliable classification performance.

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Model	Fold 1	Fold 2	Fold 3	Fold 4	Fold 5	Mean Accuracy	SD	95% CI	Precision	Recall	F1-Score
Random Forest	0.936	0.944	0.947	0.942	0.945	0.943	0.005	0.937–0.949	0.914	0.943	0.928
SVM	0.929	0.937	0.940	0.935	0.938	0.936	0.004	0.931–0.941	0.907	0.936	0.921
XGBoost	0.941	0.948	0.951	0.946	0.949	0.947	0.004	0.942–0.952	0.918	0.947	0.932
LightGBM	0.946	0.952	0.955	0.950	0.954	0.951	0.004	0.946–0.956	0.922	0.951	0.936

TABLE 4: Five-fold cross-validation performance for shape classification

LightGBM, Light Gradient Boosting Machine; SD, Standard Deviation; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The confusion matrices presented in Figure 4 demonstrate the classification performance of the four models. As observed in Figure 4d, LightGBM correctly classified 92 spherical, 209 elliptical, and 7 polygonal nanocomposites, resulting in the fewest misclassifications among the evaluated models. In comparison, most misclassifications occurred between the spherical and elliptical classes due to their similar morphological characteristics, whereas the polygonal class exhibited relatively fewer misclassifications.

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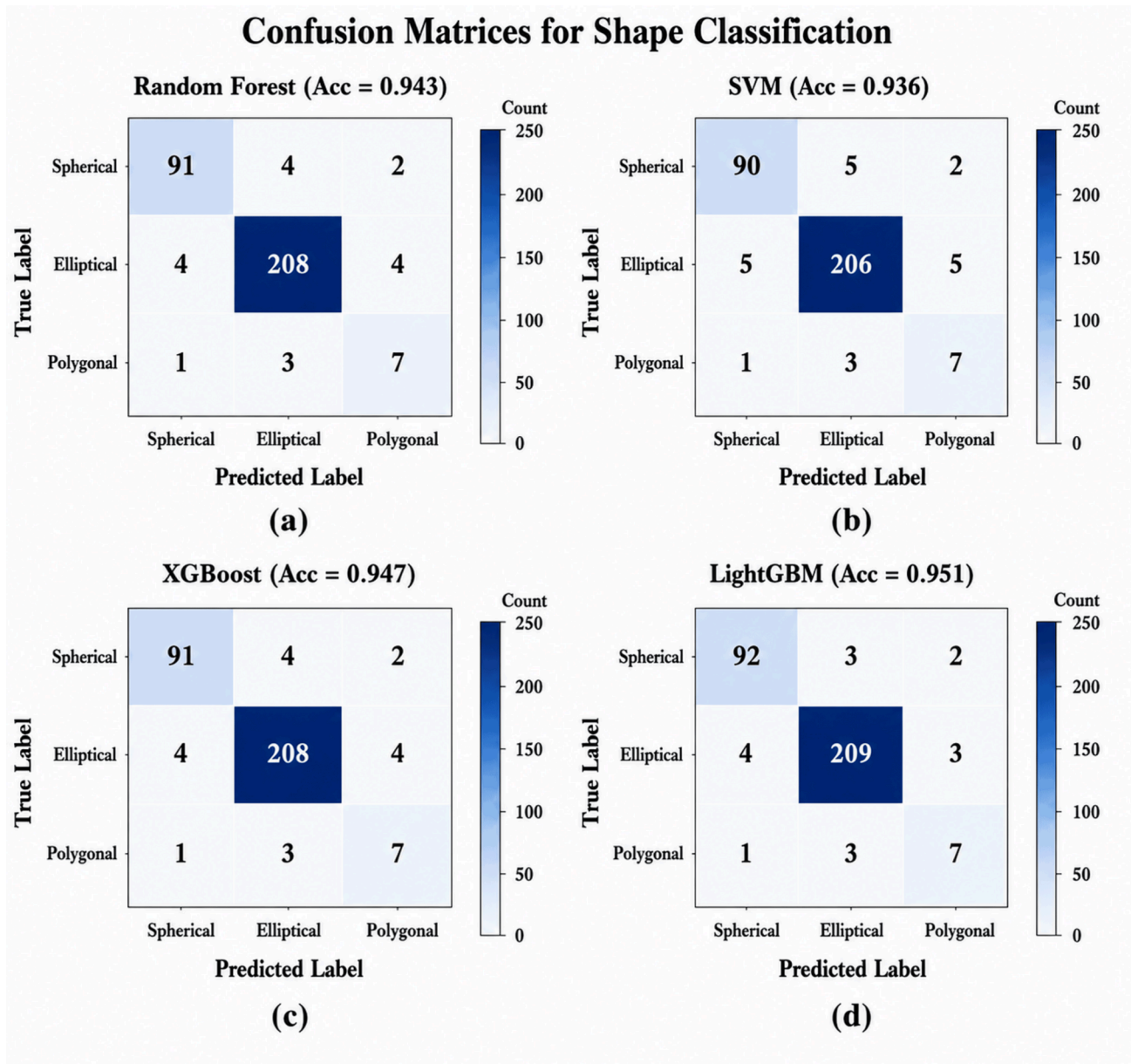


FIGURE 4: Confusion matrices of shape classification. (a) Random Forest. (b) SVM. (c) XGBoost. (d) LightGBM

LightGBM, Light Gradient Boosting Machine; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The ROC curves shown in Figure 5 further validate the discriminative capability of the classifiers. As illustrated in Figure 5d, LightGBM achieved the highest AUC values of 0.979 for spherical, 0.968 for elliptical, and 0.933 for polygonal nanocomposites. XGBoost (Figure 5c) also demonstrated excellent discrimination with AUC values of 0.966, 0.961, and 0.921, followed by Random Forest (Figure 5a) and SVM (Figure 5b). These results confirm that LightGBM provides the most accurate and reliable model for automated shape classification of molybdenum-iron oxide nanocomposites based on the extracted morphological and texture features.

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ROC Curves for Shape Classification

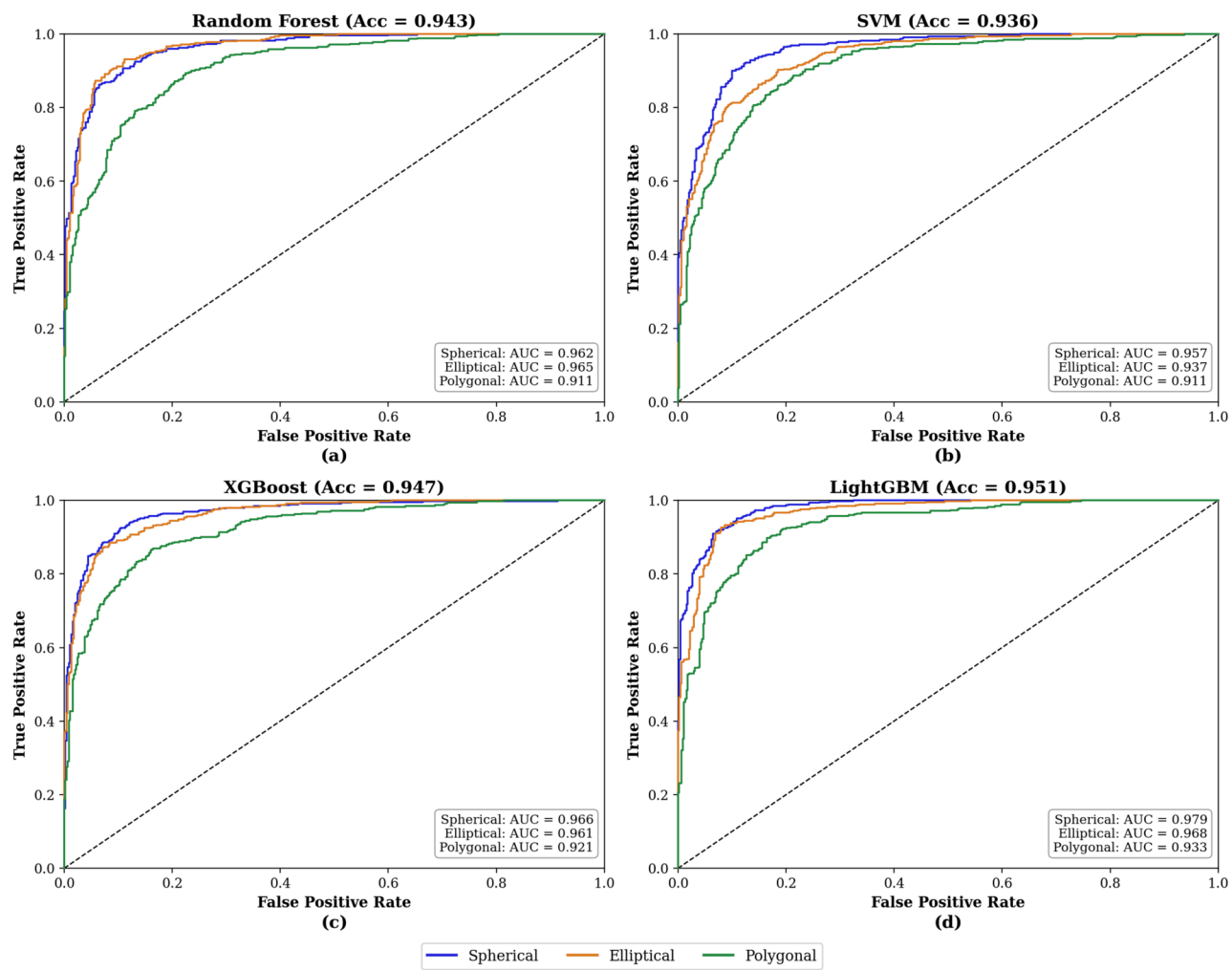


FIGURE 5: ROC curves of shape classification. (a) Random Forest. (b) SVM. (c) XGBoost. (d) LightGBM

AUC, Area Under the Curve; LightGBM, Light Gradient Boosting Machine; ROC, Receiver Operating Characteristic; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

Size classification performance

The size range classification results are presented in Table 5. Among the evaluated models, XGBoost achieved the highest mean accuracy of 96.9%, followed by LightGBM (96.1%), Random Forest (95.5%), and SVM (94.9%). XGBoost also recorded the highest precision (0.970), recall (0.969), and F1-score (0.969). The low standard deviations (0.004-0.005) and narrow 95% confidence intervals demonstrate consistent performance across all five validation folds, indicating good model stability and generalization.

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Model	Fold 1	Fold 2	Fold 3	Fold 4	Fold 5	Mean Accuracy	SD	95% CI	Precision	Recall	F1-Score
Random Forest	0.948	0.956	0.960	0.953	0.958	0.955	0.005	0.949–0.961	0.955	0.955	0.954
SVM	0.943	0.950	0.953	0.949	0.951	0.949	0.004	0.944–0.954	0.949	0.949	0.948
XGBoost	0.962	0.970	0.973	0.968	0.972	0.969	0.004	0.964–0.974	0.970	0.969	0.969
LightGBM	0.955	0.962	0.965	0.960	0.963	0.961	0.005	0.955–0.967	0.961	0.961	0.960

TABLE 5: Five-fold cross-validation performance for size classification

LightGBM, Light Gradient Boosting Machine; SD, Standard Deviation; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The confusion matrices shown in Figure 6 indicate that all classifiers correctly identified most particles, as reflected by the dominant diagonal elements. XGBoost (Figure 6c) produced the fewest misclassifications, correctly predicting 38, 210, 60, and 8 samples in the 0-50 nm, 51-100 nm, 101-150 nm, and 151-200 nm classes, respectively. Most classification errors across all models occurred between adjacent size ranges, particularly between 101-150 nm and 151-200 nm, whereas confusion between distant size classes was negligible. This pattern explains the superior performance of XGBoost compared with the other classifiers.

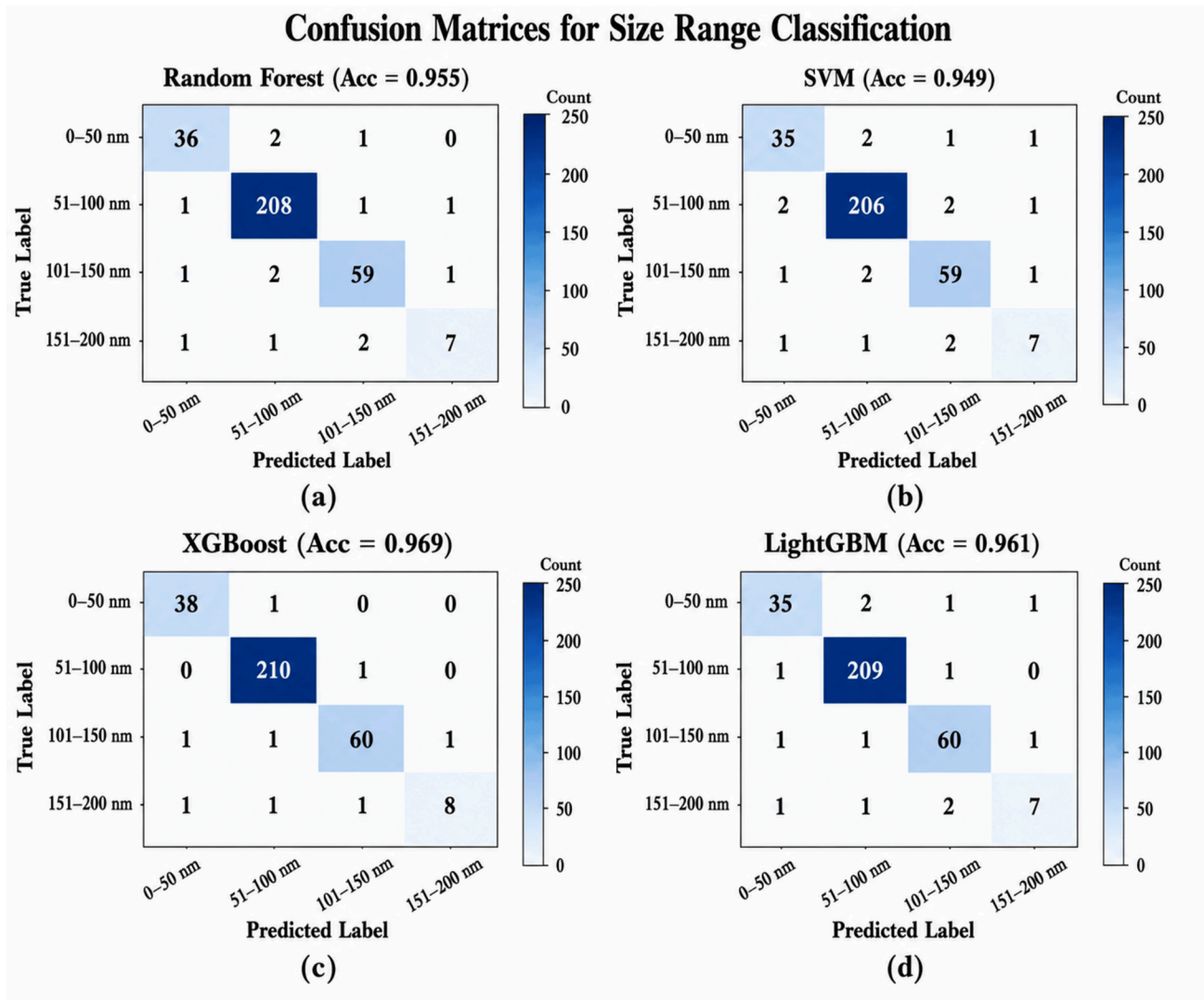


FIGURE 6: Confusion matrices of size classification. (a) Random Forest. (b) SVM. (c) XGBoost. (d) LightGBM

LightGBM, Light Gradient Boosting Machine; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The ROC curves in Figure 7 confirm the strong discriminative ability of the proposed models. XGBoost (Figure 7c) achieved the highest AUC values of 0.994, 0.990, 0.986, and 0.936 for the 0-50 nm, 51-100 nm, 101-150 nm, and 151-200 nm classes, respectively. Random Forest and LightGBM also exhibited high AUC values above 0.95 for most classes, while SVM showed comparatively lower performance, particularly for the 151-200 nm class (AUC = 0.903). Across all models, the 0-50 nm and 51-100 nm categories were classified with the highest discrimination, whereas the 151-200 nm class remained the most challenging due to overlap with neighboring size ranges. These findings are consistent with the confusion matrices presented in Figure 6 and support the superior classification capability of XGBoost for particle size prediction.

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ROC Curves for Size Range Classification

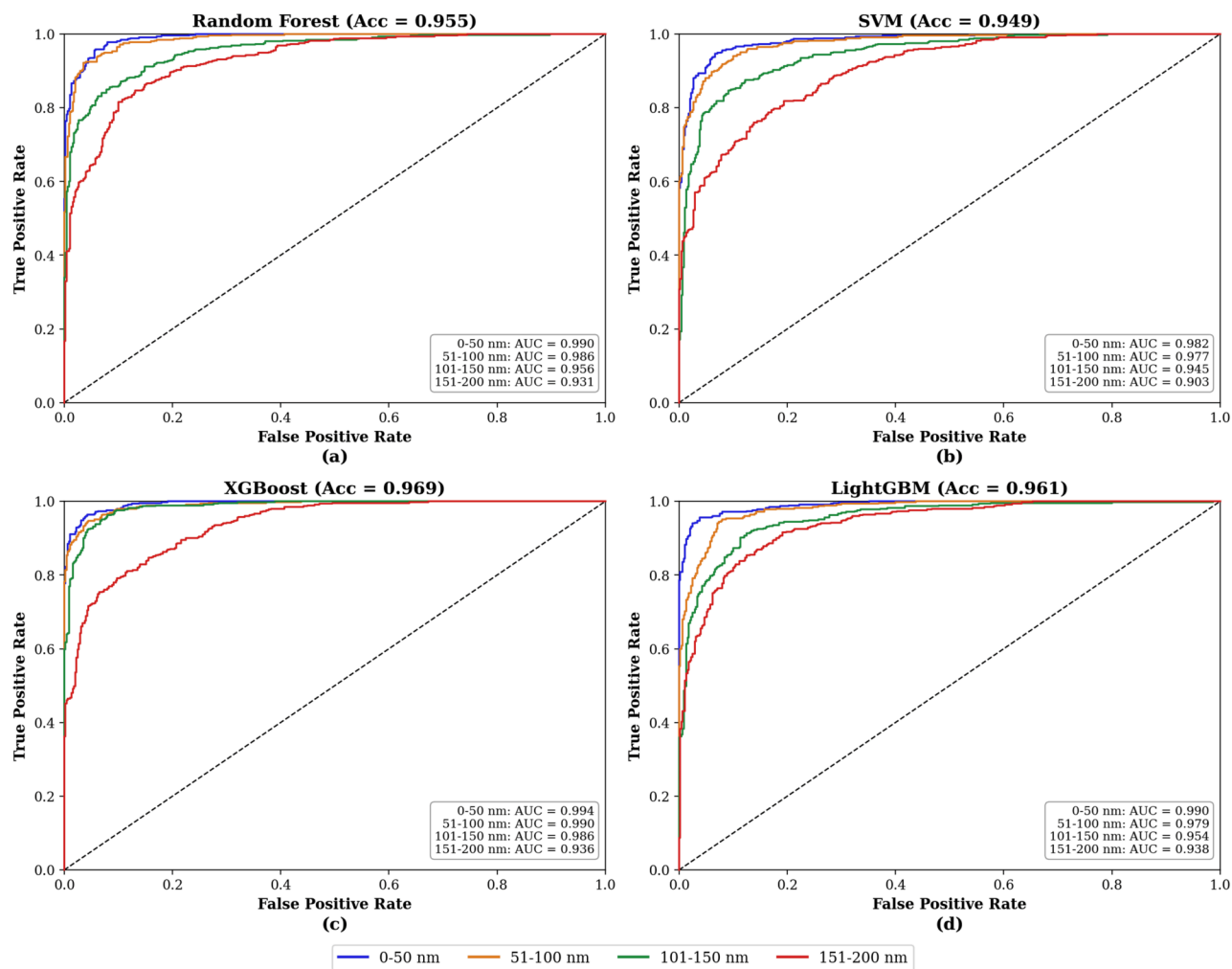


FIGURE 7: ROC curves of size classification. (a) Random Forest. (b) SVM. (c) XGBoost. (d) LightGBM

AUC, Area Under the Curve; LightGBM, Light Gradient Boosting Machine; ROC, Receiver Operating Characteristic; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

Texture classification performance

The texture classification results are summarized in Table 6. Among the evaluated classifiers, XGBoost achieved the highest mean accuracy of 94.8%, followed by LightGBM (93.4%), Random Forest (92.7%), and SVM (91.2%). XGBoost also recorded the highest precision (0.949), recall (0.948), and F1-score (0.948). The low standard deviations (0.004-0.005) and narrow 95% confidence intervals indicate stable and consistent performance across all five cross-validation folds.

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Model	Fold 1	Fold 2	Fold 3	Fold 4	Fold 5	Mean Accuracy	SD	95% CI	Precision	Recall	F1-Score
Random Forest	0.920	0.928	0.932	0.926	0.930	0.927	0.005	0.921–0.933	0.927	0.927	0.926
SVM	0.905	0.913	0.916	0.911	0.914	0.912	0.004	0.907–0.917	0.912	0.912	0.911
XGBoost	0.941	0.949	0.953	0.947	0.950	0.948	0.005	0.942–0.954	0.949	0.948	0.948
LightGBM	0.928	0.935	0.939	0.933	0.936	0.934	0.004	0.929–0.939	0.934	0.934	0.933

TABLE 6: Five-fold cross-validation performance for texture classification

LightGBM, Light Gradient Boosting Machine; SD, Standard Deviation; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The confusion matrices in Figure 8 show that all classifiers correctly identified the majority of texture classes, with most samples concentrated along the diagonal. XGBoost (Figure 8c) produced the fewest classification errors, correctly classifying 102 Smooth, 104 Moderately Rough, and 101 Rough samples. In comparison, LightGBM correctly identified 100, 103, and 100 samples for the respective classes, while Random Forest and SVM exhibited slightly more misclassifications.

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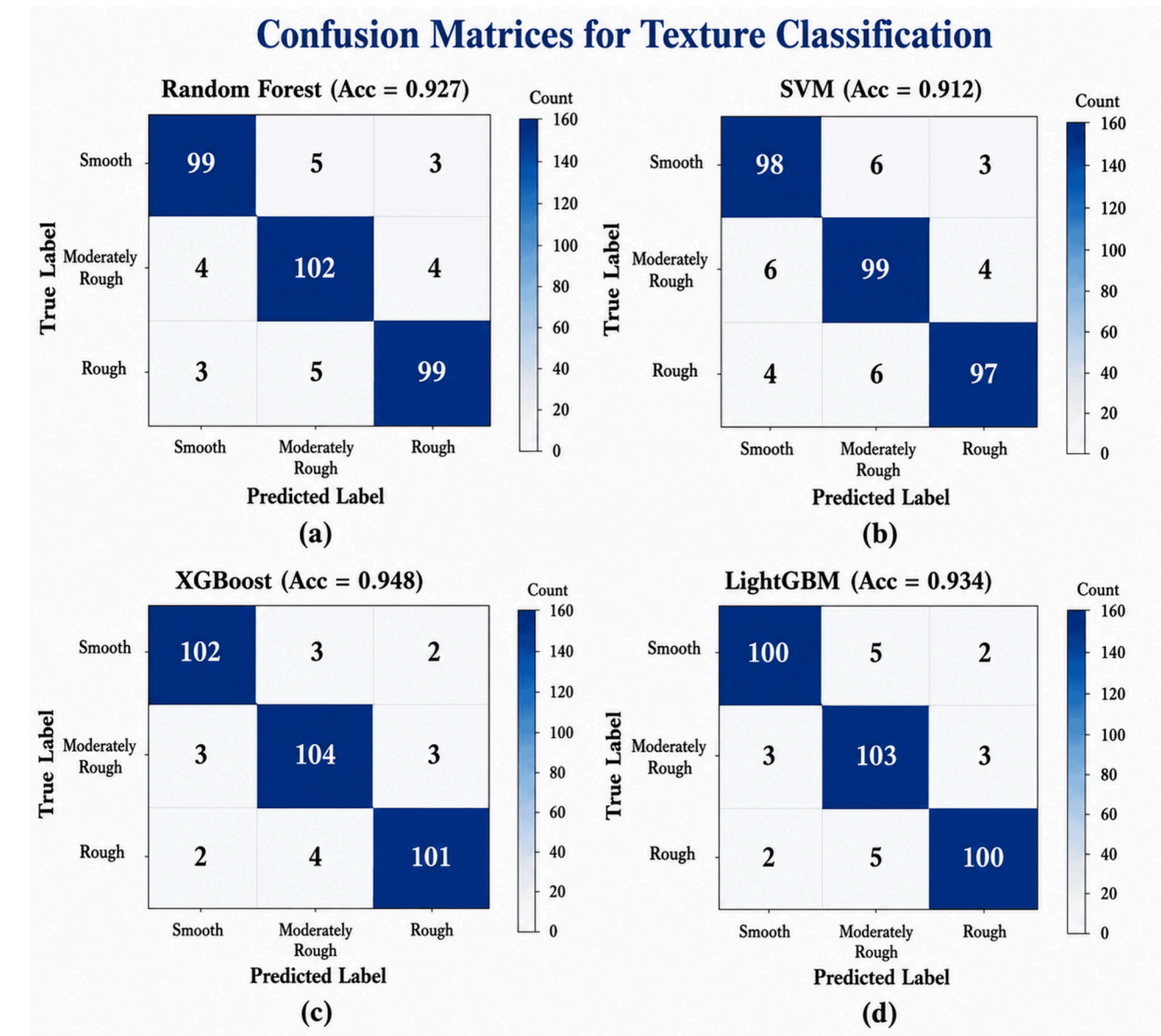


FIGURE 8: Confusion matrices of the texture classification. (a) Random Forest. (b) SVM. (c) XGBoost. (d) LightGBM
LightGBM, Light Gradient Boosting Machine; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The ROC curves presented in Figure 9 demonstrate strong discriminative performance for all classifiers. XGBoost (Figure 9c) achieved the highest class-wise AUC values of 0.987 (Smooth), 0.999 (Moderately Rough), and 0.979 (Rough), indicating excellent separation among the three texture categories. LightGBM also showed high discrimination, with AUC values of 0.968, 0.955, and 0.962, followed by Random Forest (0.969, 0.959, and 0.956). SVM produced comparatively lower AUC values (0.976, 0.950, and 0.957), consistent with its lower overall accuracy. The ROC analysis, together with the confusion matrices in Figure 8, confirms that XGBoost provides the most reliable texture classification performance among the evaluated models.

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ROC Curves for Texture Classification

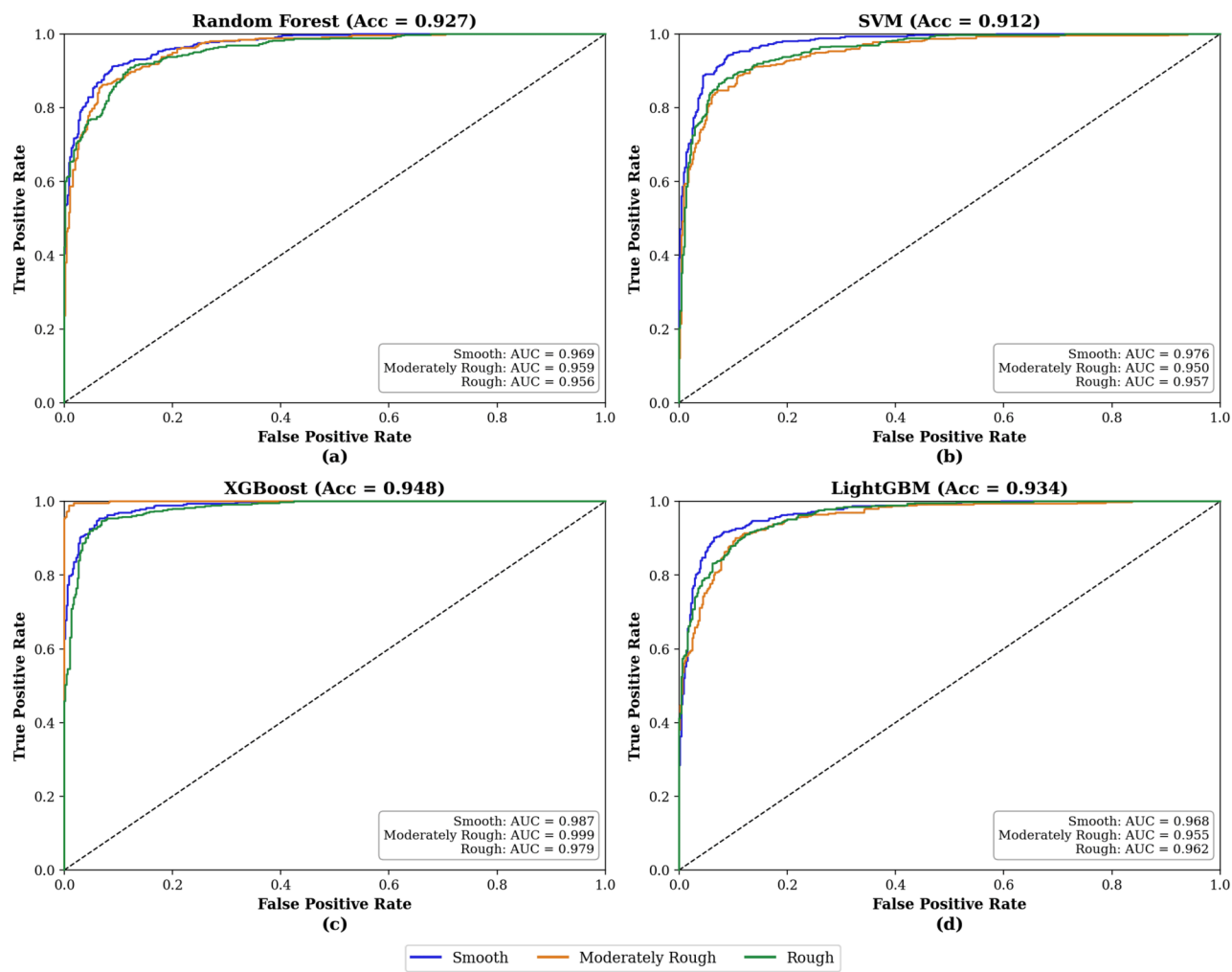


FIGURE 9: ROC curves of texture classification. (a) Random Forest. (b) SVM. (c) XGBoost. (d) LightGBM

AUC, Area Under the Curve; LightGBM, Light Gradient Boosting Machine; ROC, Receiver Operating Characteristic; SVM, Support Vector Machine; XGBoost, Extreme Gradient Boosting

The good results of gradient boosting techniques show their ability to process high-dimensional nanostructural data. These findings affirm the accuracy of the suggested framework for nanocomposite analysis through automated SEM. The findings demonstrate the suitability of gradient boosting algorithms for modeling nonlinear associations among morphological and texture descriptors.

Statistical analysis of classification performance

The statistical analysis of model performance is presented in Table 7 and provides additional evidence for the reliability of the proposed classification framework. Standard deviation values between 0.004 and 0.005, together with narrow 95% confidence intervals, indicate stable performance across the five cross-validation folds. A one-way ANOVA revealed significant differences among the evaluated machine learning models for texture, size range, and shape classification ($p <$

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0.001). The paired t-test further confirmed that the best-performing model consistently achieved significantly higher accuracy than the runner-up. More importantly, the very large Cohen's d values demonstrate that these improvements are substantial in practice rather than merely statistically significant.

Statistical Analysis	Texture Classification	Size Range Classification	Shape Classification	Interpretation
Standard Deviation	0.005	0.004	0.004	Low variability across folds indicates consistent model performance.
95% Confidence Interval	0.942–0.954	0.964–0.974	0.946–0.956	Narrow confidence intervals indicate reliable accuracy estimates.
One-way ANOVA	F = 59.996, p < 0.001	F = 20.586, p < 0.001	F = 14.055, p < 0.001	Significant differences were observed among the models.
Paired t-test (Best vs. Runner-up)	t = 69.00, p < 0.001	t = 25.30, p < 0.001	t = 17.96, p < 0.001	The best-performing model significantly outperformed the runner-up.
Cohen's d (Effect Size)	30.86 (Very large)	11.31 (Very large)	8.03 (Very large)	The improvement is statistically and practically significant.

TABLE 7: Statistical significance analysis of model performance

ANOVA, Analysis of Variance

Morphology and functional applications correlation

The nanocomposite structural characteristics play a significant role in determining the potential applications of the synthesized nanocomposites. The size distribution, with the major percentage in the range of 51-100 nm, gives the particles a high surface-to-volume ratio, which is commonly linked with increased surface reactivity and adsorption capacity. The predominance of elliptical and moderately elongated particles increases the effective surface area. The low homogeneity and high contrast values obtained from the GLCM analysis show the presence of rough and porous surfaces. These morphological and textural features are usually considered useful in several surface-dependent applications. The uneven and porous structure can be helpful for better adsorption of gas molecules and can provide smooth diffusion channels in gas-sensing applications. The heterogeneous surfaces are correlated with a high density of catalytic active sites, which may be useful in catalytic and photocatalytic reactions. Porous and non-uniform surfaces improve electrolyte penetration and ion transport, enhancing charge storage in supercapacitors and batteries. Overall,

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the combined morphological and texture analysis suggests that the synthesized nanocomposites possess structural features that can be potentially beneficial for gas sensing, catalytic, and electrochemical energy storage applications. However, further experimental investigations are needed to validate these applications.

Conclusions

This study presents a fully automated framework for the quantitative morphological and texture characterization of molybdenum-iron oxide nanocomposites from SEM images. Cellpose-based instance segmentation, feature extraction, and machine learning classification were combined into a single workflow, enabling scalable and reproducible analysis with minimal manual intervention. The segmentation framework successfully isolated 324 nanocomposites from complex SEM microstructures, highlighting the capability of deep learning for automated nanocomposite analysis. Gradient boosting methods consistently produced the strongest results, with XGBoost achieving mean accuracies of 94.8% for texture classification and 96.9% for size range classification, while LightGBM delivered the highest mean accuracy of 95.1% for shape classification. These results demonstrate that integrating deep learning with machine learning provides an effective strategy for quantitative nanocomposite characterization and supports feature-driven investigation of structure-property relationships. The present findings, however, are based on a single molybdenum-iron oxide nanocomposite dataset. Broader validation remains essential. Evaluating the framework on larger datasets, diverse SEM imaging conditions, and additional nanomaterial systems will provide stronger evidence for its robustness and enable confident generalization to wider applications.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

Concept and design: Sagar Chingali, Parashuram Bannigidad , Prabhuodeyara Gurubasavaraj

Acquisition, analysis, or interpretation of data: Sagar Chingali, Parashuram Bannigidad , Prabhuodeyara Gurubasavaraj

Drafting of the manuscript: Sagar Chingali, Parashuram Bannigidad , Prabhuodeyara Gurubasavaraj

Critical review of the manuscript for important intellectual content: Sagar Chingali, Parashuram Bannigidad , Prabhuodeyara Gurubasavaraj

Supervision: Parashuram Bannigidad , Prabhuodeyara Gurubasavaraj

Disclosures

Human subjects: All authors have confirmed that this study did not involve human participants or tissue. **Animal subjects:** All authors have confirmed that this study did not involve animal subjects or tissue. **Conflicts of interest:** In compliance with the ICMJE uniform disclosure form, all authors declare the following: **Payment/services info:** All authors have declared that no financial support was received from any organization for the submitted work. **Financial relationships:** All authors have declared that they have no financial relationships at present or within the previous three years with any organizations that might have an interest in the submitted work. **Other relationships:** All authors have declared that there are no other relationships or activities that could appear to have influenced the submitted work.

Data Availability Statements

The datasets (and/or code) supporting this study are available from the corresponding author upon reasonable request.

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