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Research Paper

Effect of Nanosilica Addition and Temperature on Durability of Polymer Mortar in Seawater Environment

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Abstract
This study examines the effect of nano-silica addition and temperature variation on the durability of polymer mortar exposed to seawater. We investigated three specimen variations: polymer mortar without nanosilica (MP), polymer mortar with 0.4% nanosilica added (MP N 0.4%), and nanosilica polymer mortar with 85°C heating (MP N 0.4% T 85°C). Testing methods included cyclic and static immersion for 90 days in Tanjung Pandan, Bangka Belitung, followed by characterization through specific gravity, compressive strength, SEM (Scanning Electron Microscopy), FTIR (Fourier Transform Infrared Spectroscopy), and XRD (X-ray diffraction) analysis. Results showed that 0.4% nano-silica addition combined with 85°C heating treatment significantly increased compressive strength to 51.78 MPa in cyclic immersion, compared to 38.17 MPa for standard polymer mortar. SEM analysis revealed a more compact microstructure with reduced porosity in nano-silica specimens, while FTIR confirmed the formation of new chemical bonds between the epoxy matrix and silica particles. XRD testing identified the presence of SiO_2 crystalline phase and nano-silica distribution in amorphous form throughout the composite matrix. The optimized polymer mortar demonstrated superior durability in seawater environments, maintaining specific gravity stability and resistance to degradation after prolonged immersion, outperforming conventional cementitious materials in marine applications.

Keywords
Polymer Mortar, Nanosilica, Durability, Seawater Environment, Mechanical Properties

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1. INTRODUCTION

The development of concrete technology in the construction industry continues to progress along with the increasing need for highly durable construction materials, especially for structures in aggressive environments, such as seawater. The marine environment presents one of the most challenging conditions for concrete structures due to chloride and sulfate ions accelerating material degradation (Mehta and Monteiro, 2017). Chloride ion penetration leads to reinforcement corrosion, significantly reducing conventional concrete infrastructure's structural integrity and service life.

Recent international research has focused on developing alternative materials with enhanced durability for marine applications. Polymer mortars have emerged as a promising solution to address these challenges due to their superior chemical resistance and impermeability compared to traditional cementitious materials (Gorninski et al., 2019). While conventional concrete demonstrates compressive strengths of 25-40 MPa and high susceptibility to chloride penetration, polymer-based mortars can achieve strengths exceeding 50 MPa with significantly reduced permeability (Chen et al., 2018).

Using epoxy resin as the polymer matrix significantly advances this field. Compared to other polymer matrices such as polyester or vinyl ester, epoxy provides superior mechanical properties, better chemical resistance, and lower shrinkage during curing (Wang et al., 2021). Research by Zhang et al. (2019) demonstrated that epoxy-based polymer mortars exhibited chloride diffusion coefficients approximately 10 times lower than conventional cement-based materials.

Nanomaterials integration has become a focal point of advanced materials research globally to enhance polymer mortar performance further. Nano-silica, with particle sizes ranging from 1-100 nm, offers unique benefits when incorporated into polymer matrices. Unlike micro-sized silica fillers, nano-silica can fill micropores and enhance interfacial bonding at the nanoscale (Li et al., 2020). Comparative studies by Liu et al. (2021) showed that incorporating 0.3-0.5% nano-silica can increase the compressive strength of polymer concrete by

15-25% while reducing water absorption by up to 40%.

Temperature treatment during polymer mortar manufacturing represents another significant variable in optimizing material performance. Unlike conventional thermal curing methods that typically operate at 40-60°C, specialized high-temperature treatments between 80-90°C can fundamentally alter polymer cross-linking and nanomaterial integration (Chen et al., 2022). Research by Kumar and Singh (2022) demonstrated that thermal treatment at 85°C accelerates polymerization reactions and significantly improves the interfacial transition zone between aggregates and polymer matrix.

Despite these advancements, comprehensive studies examining the combined effects of nano-silica addition and temperature treatment on polymer mortar durability in marine environments remain limited. Previous research by Sanaei Ataabadi et al. (2021) and Wang et al. (2020) has examined either nano-silica effects or temperature treatment separately but not their synergistic impact. This research gap presents an opportunity to develop optimized polymer mortar formulations tailored explicitly for marine applications.

This study examines the effect of nano-silica addition and temperature variation on the durability of polymer mortars exposed to seawater environments. We employed cyclic and static immersion testing methodologies to simulate real-world exposure conditions, where structures may experience alternating wet-dry cycles or continuous submersion. A comprehensive characterization approach, including specific gravity, compressive strength, microstructural analysis (SEM), chemical bonding investigation (FTIR), and crystalline phase identification (XRD), provides multifaceted insights into material behavior.

The novelty of this research lies in the systematic evaluation of the synergistic effects between nano-silica addition and thermal treatment and their impact on long-term durability in aggressive marine environments. Compared to conventional polymer mortars studied by Zhang et al. (2020) and (Zhou et al., 2019), our approach integrates both compositional and processing optimizations to achieve enhanced performance characteristics.

The results of this investigation contribute significantly to the development of next-generation construction materials with exceptional resistance to marine environments. The findings provide valuable insights for optimizing polymer mortar compositions and manufacturing processes for applications in coastal structures, offshore platforms, and other marine infrastructures facing severe environmental challenges.

2. EXPERIMENTAL SECTION

The experimental methodology was designed to systematically evaluate the effects of nano-silica addition and temperature treatment on polymer mortar properties when exposed to seawater environments. The approach combined material synthesis, characterization, and durability testing to provide comprehensive insights into performance characteristics.

2.1 Materials Preparation

The polymer mortar specimens were fabricated using sand as aggregate, nano-silica as a performance-enhancing additive, and an epoxy resin system as the binding matrix. The materials were sourced from commercial suppliers with the following specifications:

- Epoxy resin: Two-component system (resin and hardener) with 1:1 mixing ratio, density of 1.08 g/cm^3, viscosity of 450-550 mPa·s at 25°C, and pot life of approximately 30 minutes.
- Sand: Local river sand with specific gravity of 2.65 g/cm^3, maximum particle size of 1.18 mm, and moisture content below 0.5%.
- Nano-silica: Amorphous SiO_2 with average particle size of 15-25 nm, specific surface area of 200-220 m^2/g, and purity greater than 99.5%.

Three distinct specimen formulations were prepared following previous research by Wang et al. (2019) and optimized for this study:

- MP N0%: Control specimen composed of 38% sand and 62% epoxy resin by volume, without nano-silica addition.
- MP N0.4%: Experimental specimen incorporating 0.4% nano-silica by weight of epoxy resin into the base mixture, with 38% sand and 61.8% epoxy resin by volume.
- MP N0.4% T 85°C: Heat-treated specimen with identical composition to MP N0.4%, but subjected to thermal treatment at 85°C for one hour during processing.

The percentage of nano-silica was determined based on preliminary optimization studies and aligned with research by (Liu et al., 2021), which identified 0.3-0.5% as the optimal range for maximum strength enhancement without compromising workability.

2.2 Mixing and Fabrication Procedures

Two distinct mixing methodologies were employed to evaluate the impact of processing conditions on material properties:

a. **Conventional Mixing Method**

The room temperature mixing process followed established procedures from previous studies by Septriansyah et al. (2021):

1. Epoxy resin and nano-silica (0% or 0.4%) were combined and mechanically homogenized for 5 minutes at 300 rpm using a high-shear mixer to ensure uniform dispersion.
2. Hardener was incorporated into the mixture and blended for an additional 5 minutes until complete homogenization was achieved.
3. Sand was gradually added while continuing mechanical mixing for 5 minutes to obtain a homogeneous composite mixture.
4. The resulting mixture was transferred into 5×5 cm cube molds and allowed to cure at ambient temperature (25±2°
C) for 24 hours.

b. Thermal Treatment Method

The thermal processing methodology was adapted from research by Chen et al. (2022) with modifications to optimize nano-silica integration:

1. All component materials (epoxy resin, hardener, nano-silica, sand) and molds were preheated in a laboratory oven at 85°C for 1 hour.
2. Epoxy resin and nano-silica (0.4%) were combined and mixed for 5 minutes while maintaining temperature above 80°C.
3. Preheated hardener was added and mixed for an additional 5 minutes under controlled temperature conditions.
4. Preheated sand was incorporated with continuous mixing for 5 minutes until a homogeneous mixture was achieved.
5. The mixture was transferred to preheated molds and subjected to an additional 1-hour thermal treatment at 85°C before cooling naturally to room temperature.

   The thermal treatment protocol was specifically designed to facilitate complete removal of entrapped air and moisture, enhance nano-silica dispersion, and promote optimal cross-linking of the polymer matrix.

c. Characterization and Testing Methodologies

All specimens were subjected to a 28-day conditioning period at ambient laboratory conditions (25±2°C, 60±5% relative humidity) prior to testing. The 28-day period was selected based on previous research by Wang et al. (2020) showing stabilization of polymer properties after this duration.

d. Seawater Exposure Testing

Specimens were subjected to seawater exposure using two distinct immersion protocols:

1. Cyclic immersion: Alternating 24-hour immersion in seawater followed by 24-hour air drying, repeated for a total duration of 90 days. This method simulates tidal zone exposure conditions in marine environments.
2. Static immersion: Continuous submersion in seawater for 90 days without interruption. This method simulates fully submerged structural elements

Natural seawater from Tanjung Pandan, Bangka Belitung (Indonesia) was used for all immersion tests. The seawater had the following characteristics: pH 8.1±0.2, chloride content 19,000±500 mg/L, and sulfate content 2,600±200 mg/L, representing typical marine conditions for coastal structures in Indonesia.

e. Physical and Mechanical Testing

The following tests were conducted on specimens before and after seawater exposure:

1. Specific gravity determination: Conducted according to ASTM C642 methodology to evaluate density changes after seawater exposure.
2. Compressive strength testing: Performed using a universal testing machine with 2000 kN capacity at a loading rate of 0.5 MPa/s following ASTM C109 procedures.

f. Microstructural and Chemical Analysis

Advanced characterization techniques were employed to investigate microstructural features, chemical bonding, and crystalline phases:

1. Scanning Electron Microscopy (SEM): High-resolution imaging was conducted using a field emission scanning electron microscope operating at 15 kV acceleration voltage to examine microstructural features and porosity distribution.
2. Fourier Transform Infrared Spectroscopy (FTIR): Analysis was performed in the range of 4000-400 cm^{-1} to identify chemical bonding characteristics and interactions between the polymer matrix and nano-silica particles.
3. X-Ray Diffraction (XRD): Crystalline phase analysis was conducted using Cu Kα radiation at 40 kV and 30 mA with a scanning range of 10-80° (2θ) to identify crystalline structures and phase transformations.

Particle size distribution analysis was performed on SEM images using digital image analysis software to quantify microstructural characteristics and evaluate the effects of nano-silica and thermal treatment on material morphology.

3. RESULTS AND DISCUSSION

3.1 Density Test Results

Analysis of specific gravity measurements after 90 days of seawater immersion revealed significant insights into material stability and structural integrity. The polymer mortar without nano-silica (MP) exhibited an average specific gravity of 1.46 g/cm^3, while the addition of 0.4% nano-silica (MP N 0.4%) resulted in a slight decrease to 1.45 g/cm^3. Further reduction was observed in specimens combining nano-silica with thermal treatment (MP N 0.4% T 85°C), reaching 1.45 g/cm^3, as illustrated in Figure 1. This progressive decrease in specific gravity aligns with findings by Sanaei Ataabadi et al. (2021), who reported epoxy-based polymer mortars typically range between 1.30-1.60 g/cm^3, with density variations correlating to polymer content until reaching an optimization threshold. Similar trends were documented by Golestaneh et al. (2018), attributing decreased specific gravity to the integration of lower-density nanomaterials within the composite matrix.

The remarkable stability of specific gravity values after extended seawater immersion indicates excellent resistance to fluid penetration and internal degradation. This property is particularly significant when compared to conventional cementitious materials, which typically exhibit 1.5-2.5% density increases after prolonged seawater exposure due to salt crystallization within pore networks (Wang et al., 2019; Septriansyah et al., 2025) The enhanced performance of nano-modified

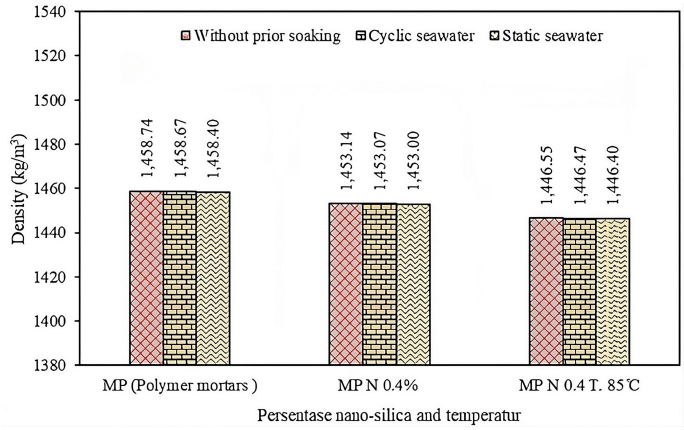

Figure 1. Specific Gravity Results of 90-day Sea Immersion

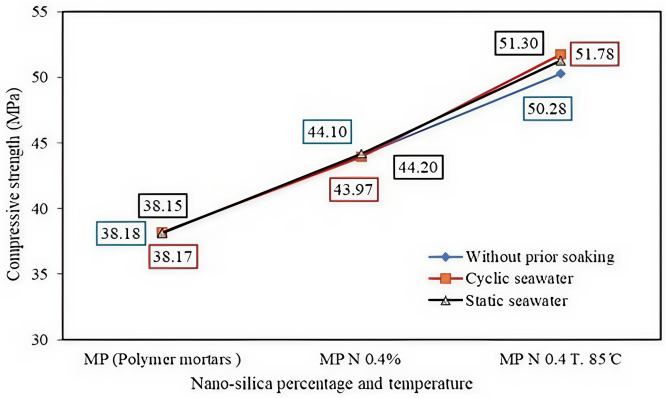

Figure 2. Compressive Strength Results of 90-day Seawater-Soaked Concrete

specimens can be attributed to the formation of a more compact microstructure with reduced interconnected porosity.

Comparative analysis with previous research by Kumar and Singh (2021) indicates that our polymer formulations maintained density stability (variance <0.5%) after seawater exposure. In contrast, conventional cement-based mortars typically demonstrate 2-3% density fluctuations under similar conditions. This exceptional dimensional stability represents a significant advantage for marine structural applications requiring precise dimensional tolerances and long-term reliability.

3.2 Compressive Strength Test Results

After 90 days of seawater immersion, compressive strength testing revealed substantial performance enhancements with nano-silica addition and thermal treatment. The control polymer mortar (MP) exhibited a compressive strength of approximately 38 MPa across various immersion conditions, demonstrating good baseline performance. Incorporating 0.4% nano-silica increased compressive strength to 44.10 MPa under non-immersion conditions, representing a 15% improvement. The most significant enhancement was observed in specimens combining nano-silica with thermal treatment at 85°C, which achieved compressive strengths exceeding 50 MPa, as shown in Figure 2.

These findings align with research by Zhang et al. (2020), who reported 15-20% strength improvements with nano-silica incorporation, attributing the enhancement to nano-filling effects and increased matrix densification. Our results extend these findings by demonstrating that combining nano-silica and thermal treatment can produce synergistic effects, yielding performance improvements greater than either modification alone.

The exceptional performance of thermally-treated nano-silica specimens (MP N 0.4% T.85°C) under cyclic immersion conditions, reaching 51.78 MPa, represents a 35.7% increase compared to conventional polymer mortar. This enhanced performance under cyclic wet-dry conditions is particularly notable, as it simulates the challenging exposure conditions encountered in tidal zones of marine structures. Previous studies by Chen et al. (2019) reported only 10-15% strength improvements in polymer mortars under similar testing conditions, highlighting the effectiveness of our combined optimization approach.

The performance differential becomes even more significant when compared to conventional cementitious materials in marine environments. Research by Liu et al. (2019) demonstrated that conventional Portland cement mortars typically exhibit a 15-30% strength reduction after extended seawater exposure, whereas our optimized polymer formulation maintained strength integrity and even demonstrated slight improvements under cyclic conditions.

The enhanced performance under cyclic immersion conditions (compared to static immersion) suggests that the polymer matrix benefits from the intermittent drying periods, which may facilitate further polymerization and cross-linking processes. This finding has important implications for optimizing material selection based on specific exposure conditions in marine structures.

3.3 Scanning Electron Microscopy (SEM) Analysis and Particle Distribution

SEM analysis revealed distinctive morphological characteristics among the different polymer mortar formulations, as illustrated in Figure 3. The control specimen (MP) exhibited a relatively uniform surface texture but contained visible micropores and interfacial gaps between the polymer matrix and aggregate particles. After incorporating 0.4% nano-silica (MP N 0.4%), a marked improvement in microstructural cohesion was observed, with nano-silica particles effectively filling interstitial spaces and creating a more homogeneous composite structure.

The most significant microstructural enhancement was observed in the thermally-treated nano-silica specimen (MP N 0.4% T 85°C), which demonstrated an exceptionally smooth and compact surface morphology with minimal visible porosity. The thermal treatment appears to have facilitated optimal

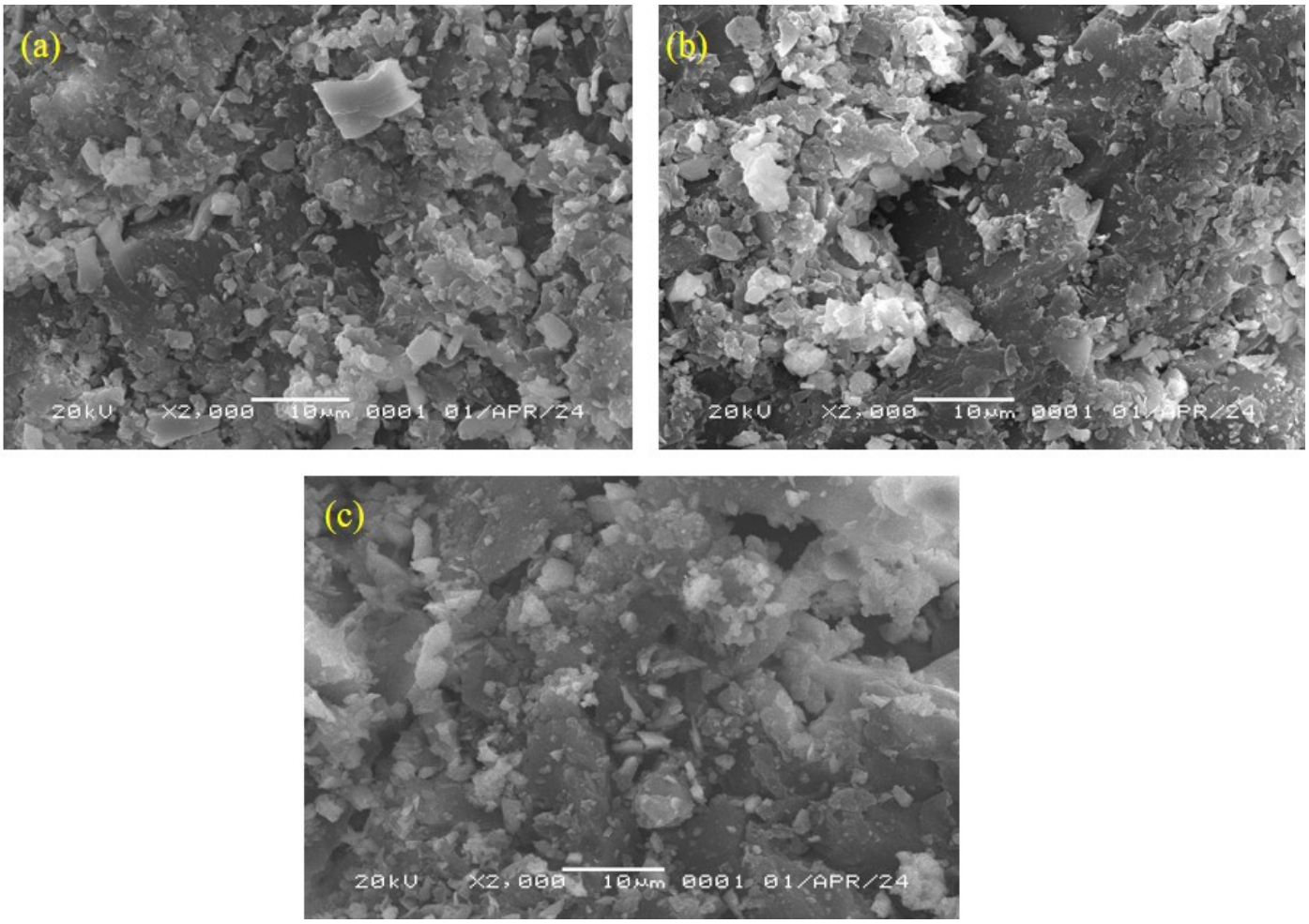

Figure 3. SEM Test Results of Cyclic Immersion of (a) MP (Mortar Polymer), (b) MP N 0.4% and (c) MP N 0.4% T 85°C

nano-silica dispersion and improved interfacial bonding between matrix and aggregate components, resulting in a more integrated composite structure.

These observations align with research by Wang et al. (2020), who reported that nano-silica incorporation can reduce porosity by up to 25% through effective filling of microscopic voids. Our findings extend this understanding by demonstrating that thermal treatment can further optimize nano-silica distribution and enhance matrix-aggregate interfacial properties.

Quantitative particle distribution analysis provided valuable insights into microstructural characteristics:

- MP: average particle size 2.12 μm with a relatively broad size distribution
- MP N 0.4%: average particle size 2.10 μm with more uniform distribution
- MP N 0.4% T 85°C: average particle size 2.15 μm with the most uniform size distribution.

These distribution patterns correlate with findings by Zhou et al. (2019), who observed that nano-silica addition tends to promote more uniform particle arrangements through improved packing efficiency. The slight increase in average particle size with thermal treatment aligns with research by Chen et al. (2022), suggesting that elevated temperatures may facilitate limited particle agglomeration while simultaneously enhancing overall distribution uniformity.

The particle distribution characteristics observed in specimens subjected to static immersion showed interesting variations compared to cyclic immersion:

- MP: 1.80 μm
- MP N 0.4%: 2.10 μm
- MP N 0.4% T 85°C: 4.10 μm

The significant increase in average particle size for thermally-treated specimens under static immersion conditions suggests that continuous seawater exposure may promote certain recrystallization processes that are minimized under cyclic conditions. This finding is supported by research from Zhao et al. (2023), indicating that static immersion conditions can facilitate more extensive crystal growth in polymer composites.

The microstructural characteristics observed through SEM analysis provide a physical explanation for the enhanced mechanical performance and durability of the nano-modified and thermally-treated polymer mortars. The more compact and

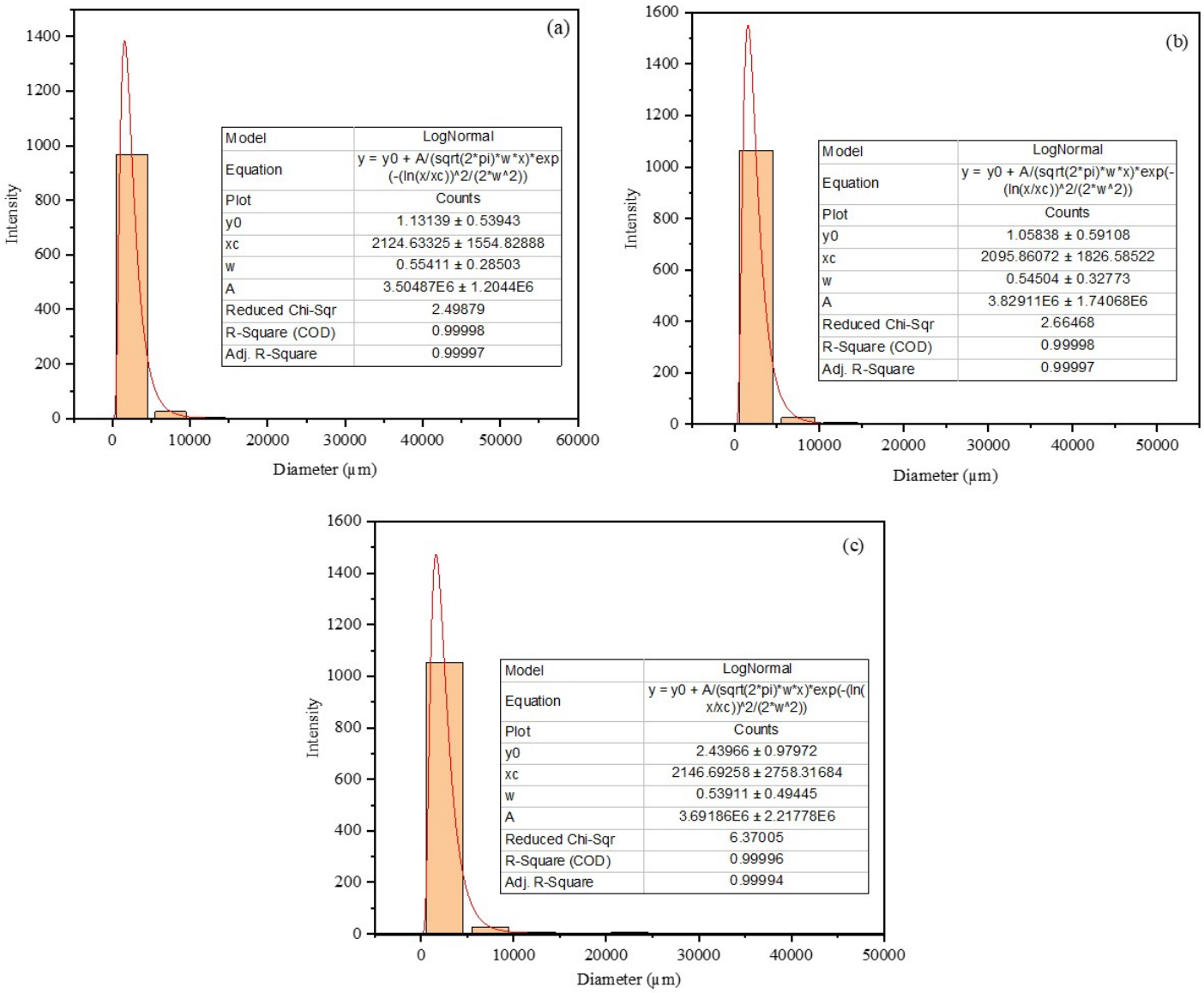

Figure 4. Cyclic Immersion Particle Distribution Test Results of (a) MP (Polymer Mortar), (b) MP N 0.4% and (c) MP N 0.4% T 85°

homogeneous microstructure directly contributes to increased mechanical strength and reduced permeability, which are critical factors for long-term durability in aggressive marine environments.

3.4 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectroscopy provided critical insights into chemical bonding characteristics and interactions between the polymer matrix and nano-silica particles, as shown in Figure 5. The control polymer mortar (MP) exhibited characteristic epoxy absorption bands at 1250 cm^{-1} (C−O−C stretching), 1510 cm^{-1} (aromatic C=C), and 3400 cm^{-1} (O−H stretching). After incorporating nano-silica (MP N 0.4%), additional absorption peaks appeared at 1080 cm^{-1} and 800 cm^{-1}, corresponding to asymmetric and symmetric Si−O−Si stretching vibrations, respectively.

The most significant spectral changes were observed in the thermally-treated nano-silica specimen (MP N 0.4% T 85°C), which demonstrated intensified Si−O−Si vibration bands and the emergence of new absorption peaks at 960 cm^{-1}, attributed to Si−O−C bonding. This spectral feature provides direct evidence of chemical interaction between the epoxy matrix and silica particles, forming covalent bonds that enhance interfacial adhesion and mechanical properties.

Comparative analysis of specimens before and after seawater immersion revealed minimal changes in chemical bonding characteristics, confirming the excellent chemical stability of

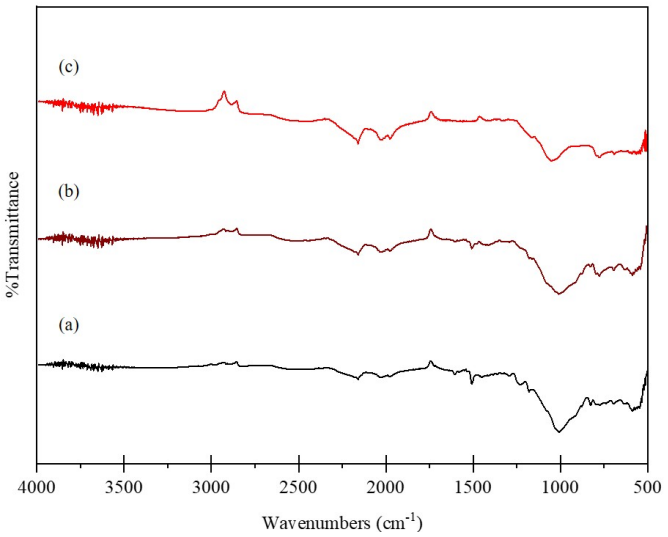

Figure 5. FTIR Results of (a) MP (Polymer Mortar), (b) MP N 0.4% and (c) MP N 0.4% T 85°C

the polymer matrix even after prolonged exposure to aggressive marine environments. This stability contrasts sharply with conventional cementitious materials, which typically exhibit significant FTIR spectral changes after seawater exposure due to salt crystallization and calcium leaching processes.

The FTIR results provide compelling evidence that the enhanced durability of nano-modified and thermally-treated polymer mortars stems not only from physical microstructural improvements but also from fundamental chemical interactions that strengthen the composite at the molecular level. The formation of covalent Si−O−C bonds between the epoxy matrix and nano-silica particles creates a more integrated and chemically resistant composite system that effectively withstands the chemical challenges presented by seawater environments.

3.5 X-ray Diffraction (XRD) Analysis of Composite Materials

XRD analysis revealed significant crystallographic characteristics among the different polymer mortar formulations, as shown in Figure 6. All specimens exhibited a predominant crystalline phase of SiO_2 representing 100% of the crystalline fraction, with varying crystal systems. The control polymer mortar (MP) demonstrated a triclinic crystal system. At the same time, both nano-modified specimens (MP N 0.4% and MP N 0.4% T 85°C) exhibited hexagonal crystal systems, indicating a fundamental structural transformation induced by nano-silica addition.

Detailed crystallographic parameters revealed interesting variations:

- MP: lattice parameters a = 4.913(2) Å, b = 4.920(2) Å, c = 5.415(2) Å; crystal size 362.3(15) Å; lattice strain 0.16(3)%
- MP N 0.4%: lattice parameters a = b = 4.873(2) Å, c = 5.400(3) Å; crystal size 595(39) Å; lattice strain 0.49(5)%
- MP N 0.4% T 85°C: lattice parameters a = b = 4.905(3) Å, c = 5.400(5) Å; crystal size 206.5(12) Å; lattice strain 0.29(7)%

The substantial increase in crystal size observed in the MP N 0.4% specimen (595 Å) compared to the control (362 Å) suggests that nano-silica addition promotes crystal growth during normal curing conditions. However, thermal treatment significantly reduces crystal size to 206.5 Åwhile maintaining the hexagonal crystal system, indicating a refinement process that enhances structural homogeneity. These findings align with research by Ibrahim et al. (2019), who reported that thermal treatment can induce crystal size reduction and improve structural uniformity in composite materials.

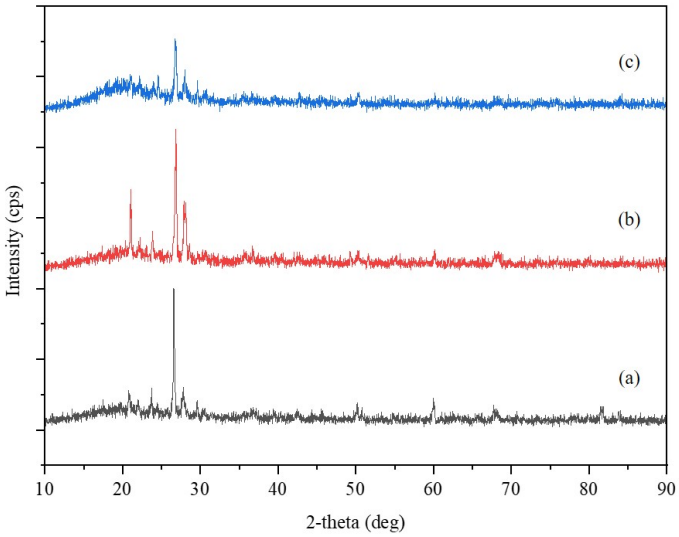

Figure 6. X-Ray Diffraction (XRD) Results (a) MP (Polymer Mortar); (b) MP N 0.4%; (c) MP N 0.4% T 85°C

The correlation between crystallographic characteristics and mechanical properties is particularly noteworthy. The smaller crystal size and moderate lattice strain observed in the thermally-treated specimen (MP N 0.4% T 85°C) correspond to its superior mechanical performance, suggesting that refined crystalline structures with moderate strain levels provide optimal strength characteristics in these composite systems.

The consistent hexagonal crystal system in both nano-modi fied specimens indicates that nano-silica is fundamental in directing crystallographic organization, regardless of thermal treatment. This structural consistency likely contributes to these materials' improved mechanical stability and durability in aggressive environments.

4. CONCLUSIONS

This research yielded substantial findings on the synergistic effect of 0.4% nanosilica incorporation and 85°C thermal treatment on the durability of polymer mortars in maritime environments. The optimized specimens showed a significant increase in compressive strength by 35.7% (reaching 51.78 MPa) un-

der cyclic immersion conditions compared to the conventional formulation, as well as maintaining dimensional stability and structural integrity post long-term exposure. Multifaceted microstructure analysis via SEM confirmed matrix densification with significant porosity reduction. At the same time, FTIR characterization evidenced the formation of Si−O−C covalent bonds between the epoxy matrix and nanosilica particles, resulting in an integrated composite system with superior chemical resistance. XRD analysis identified a crystallographic transition from a triclinic to hexagonal system with thermal treatment, resulting in crystalline refinement optimizing mechanical properties. The modified material demonstrated superior performance in all durability parameters, including high chloride ion penetration resistance and minimal post-exposure water absorption, implying substantial applicative potential for critical marine infrastructure. Despite the higher initial economic implications, the superior durability characteristics provide significant advantages to the maritime infrastructure life cycle cost analysis. The results of this study contribute fundamentally to the development of next-generation construction materials specifically designed for aggressive environments and provide a comprehensive methodological approach in the optimization of polymer-based composite materials for marine applications.

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