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Development of Multifunctional PVA-GO-TiO₂ Polymer Nanocomposites for Industrial Durability and Environmental Remediation

Qudsia Khadimzada

Department of Electrical and Computer Engineering, Basra Institute of Business Technology, Iraq
qudsia.khadimzada@bibt-iq.org

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Abstract

Polymer nanocomposites combine a continuous polymer phase with nanoscale fillers to create materials whose mechanical, thermal, barrier, electrical, and surface properties can be tailored at low filler loading. This methodology paper proposes a reproducible route for synthesizing and characterizing multifunctional polymer nanocomposites intended for both industrial and environmental applications. A crosslinked poly(vinyl alcohol) (PVA) matrix is selected as a model polymer because it can be processed from water and readily interacts with oxygen-containing nanofillers. Graphene oxide (GO), titanium dioxide (TiO₂), and a GO-TiO₂ hybrid are used as representative carbonaceous, metal-oxide, and hybrid nanofillers. The experimental design compares neat PVA with three nanocomposite formulations prepared by controlled dispersion, solution blending, casting, drying, and thermal crosslinking. Structure and dispersion are evaluated by Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS). Thermal stability, tensile properties, water uptake, and methylene-blue removal are measured to link morphology with industrial durability and environmental remediation performance. Literature-calibrated illustrative results predict that the hybrid nanocomposite can provide the best balance of tensile strength, thermal stability, dimensional integrity, and dye-removal efficiency when agglomeration is controlled. The framework is designed to support future laboratory validation and can be adapted to other polymer matrices, nanoclays, carbon nanomaterials, and metal-oxide fillers.

Introduction

Polymer nanocomposites are a class of multiphase materials in which at least one dispersed component possesses nanoscale dimensions. The very high surface-to-volume ratio of nanoscale fillers creates an extended polymer-filler interphase, so property changes can be obtained at substantially lower filler fractions than in conventional microcomposites. Early work on polymer-layered silicates

demonstrated that intercalation and exfoliation strongly influence stiffness, gas barrier behavior, thermal response, and flammability [1,2]. Subsequent analyses emphasized that the practical benefit of a nanofiller depends not only on its intrinsic properties but also on dispersion, aspect ratio, interfacial adhesion, orientation, and the processing history of the polymer matrix [3,4].

Industrial interest in polymer nanocomposites arises from the ability to reduce mass while increasing modulus, heat resistance, barrier performance, wear resistance, electrical functionality, or flame retardancy. Surface functionalization is often required because many inorganic nanoparticles are intrinsically incompatible with organic polymers; tailored surface chemistry can suppress agglomeration and promote load transfer across the interface [5]. Carbon nanotubes, graphene-family materials, nanoclays, silica, TiO_2 , ZnO , and related fillers have therefore been incorporated into thermoplastics, thermosets, elastomers, and water-processable polymers for packaging, coatings, membranes, structural parts, electronics, and sensing [6].

Environmental applications create a second design requirement. The nanocomposite must remain mechanically stable during water contact while exposing sufficient active surface for adsorption, photocatalysis, membrane separation, or antimicrobial action. Polymer-supported nanofillers are attractive because the polymer enables handling and recovery of particles that would otherwise be difficult to separate after treatment. Studies have shown that polymer/clay systems can remove organic pollutants [8], while PVA-based nanocomposites can be engineered for membrane separation and pollutant adsorption [10,11]. These findings motivate a methodology that evaluates structural and environmental performance in the same specimen set rather than treating them as unrelated objectives.

The present work develops such a methodology using PVA as a model matrix, GO as a high-aspect-ratio oxygenated carbon nanofiller, TiO_2 as a chemically stable metal-oxide nanofiller, and a GO- TiO_2 hybrid as a multifunctional system. The central hypothesis is that controlled hybridization can improve stress transfer and thermal stability while simultaneously providing accessible adsorption/photocatalytic sites. The study is intentionally designed as a methodology paper: it specifies material preparation, characterization, calculations, quality controls, and an analysis plan, while the numerical result values are clearly identified as literature-calibrated illustrative values that must be replaced by actual measurements when the protocol is executed.

Literature Review

Alexandre and Dubois [1] established the foundations of polymer-layered silicate nanocomposites and showed that nanoscale dispersion can produce large property gains when the silicate galleries are intercalated or

exfoliated. Ray and Okamoto [2] later linked preparation route and processing conditions to final morphology, emphasizing melt intercalation, solution methods, and in-situ polymerization. Pavlidou and Papaspyrides [3] consolidated evidence that dispersion state, clay modification, and polymer chemistry determine mechanical, thermal, and barrier behavior. Paul and Robeson [4] broadened the nanocomposite framework by discussing nanoscale reinforcement, percolation, transport, and property enhancement across diverse polymer systems.

Interfacial chemistry emerged as a major control variable. Kango et al. [5] reviewed surface-modification strategies for inorganic nanoparticles and showed why functional groups, coupling agents, and compatibilizers can improve dispersion and polymer-particle bonding. Bhattacharya [6] compared carbon nanotubes, graphene, and nanoclay and highlighted the different advantages of one-dimensional, two-dimensional, and layered fillers. For water-processable systems, Sharma et al. [7] reported that processing conditions affect the structural and mechanical response of graphite- and graphene-oxide-reinforced PVA, illustrating the importance of filler distribution and polymer-filler interaction.

Environmental remediation provides a direct application route for such interfacial control. Ramírez-Gómez et al. [8] synthesized polymer/clay nanocomposites for atrazine removal, demonstrating that the polymer matrix can support active sorption sites while retaining a recoverable solid form. Kausar [9] reviewed polymer/nanoclay systems combined with carbonaceous nanofillers and identified environmental, construction, aerospace, and functional-material applications. Nigiz [10] showed that graphene nanoplate incorporation can modify PVA crystallinity, mechanical strength, thermal behavior, morphology, and gas-separation performance. El-Shamy [11] developed PVA-based nanocomposite adsorbents for methylene-blue removal, supporting the use of dye removal as a practical environmental performance metric.

Recent pre-2022 reviews strengthen the rationale for the proposed matrix/filler selection. Feldman [12] summarized PVA nanocomposites containing oxides, graphene derivatives, nanoclays, and other nanofillers and emphasized their engineering versatility. Agboola et al. [13] reviewed polymer nanocomposites for membranes and adsorbents in water treatment and gas separation, demonstrating that mechanical integrity and mass-transfer performance must be evaluated together.

Ibrahim et al. [14] reviewed graphene-based nanocomposite synthesis and characterization and emphasized that processing route and dispersion state directly affect mechanical response. Abulyazied and Ene [15] similarly showed that nanoclay-reinforced polymers depend strongly on preparation method, filler

concentration, and interphase quality. Collectively, the literature identifies a consistent research need: a unified protocol should connect synthesis conditions, nanofiller dispersion, structure, bulk durability, and environmental function using one controlled formulation series.

Table 1. Selected literature supporting the proposed methodology

Ref.	System / focus	Main methodological implication
[1–4]	Layered silicates and general PNC theory	Dispersion, interphase, aspect ratio, and processing govern property gains.
[5]	Surface-modified inorganic nanoparticles	Surface chemistry is required to limit agglomeration and improve adhesion.
[6,14]	Graphene/CNT/clay nanofillers	Nanofiller geometry and dispersion affect mechanical and multifunctional behavior.
[7,10,12]	PVA-based nanocomposites	Water-processable PVA supports carbon, oxide, and clay nanofillers.
[8,11,13]	Environmental adsorption / separation	Nanocomposites can combine recoverability with pollutant-removal functionality.
[15]	Nanoclay-reinforced polymers	Preparation route and filler loading must be controlled systematically.

Table 2. Proposed formulation matrix

Code	PVA	GO	TiO ₂	Citric acid*	Purpose
P0	100	0	0	10	Neat crosslinked control
P1	100	0.5	0	10	Carbon nanofiller effect
P2	100	0	2.0	10	Metal-oxide effect
P3	100	0.5	2.0	10	Hybrid multifunctional nanocomposite

Methodology

1. Materials and experimental design

The model polymer is fully hydrolyzed PVA (molecular weight approximately 85,000–124,000 g mol⁻¹). Graphene oxide with lateral dimensions in the submicrometre-to-few-micrometre range and anatase-rich TiO₂ nanoparticles of approximately 20–30 nm is selected as nanofillers. Citric acid is used as a low-toxicity crosslinking agent and deionized water as the principal processing medium. Four formulations are prepared so that the effect of each nanofiller and the hybrid combination can be separated. Filler contents are expressed as weight percent relative to dry PVA. Each formulation is prepared in three independent batches to enable statistical analysis.

2. Step-by-step synthesis protocol

Step 1 – PVA solution preparation. Dissolve PVA in deionized water to obtain a 10 wt% solution. Heat to 85–90 °C under mechanical stirring for approximately 2 h until the solution becomes

homogeneous and free of visible granules. Cool to 40–45 °C before nanofiller addition.

Step 2 – GO dispersion. Disperse the required GO mass in deionized water using probe or bath ultrasonication for 30 min. Keep the dispersion temperature below 35 °C to minimize thermal damage and excessive reduction of oxygen-containing groups.

Step 3 – TiO₂ dispersion. Prepare a separate TiO₂ suspension by high-shear mixing followed by 20–30 min ultrasonication. For the hybrid specimen, add the TiO₂ suspension slowly to the GO dispersion and mix for a further 15 min so that oxide particles are distributed around the GO sheets.

Step 4 – Polymer–nanofiller blending. Add each nanofiller dispersion dropwise to the PVA solution while stirring at 600–800 rpm. Continue mixing for 60 min and then sonicate the final mixture for 10 min to remove soft agglomerates. The neat PVA control is subjected to the same thermal and mixing history.

Step 5 – Crosslinking and casting. Add citric acid at 10 parts per 100 parts PVA and mix for 15 min.

Degas the mixture under mild vacuum, cast a constant mass onto level glass plates or PTFE dishes, and dry at 45–50 °C for 24 h.

Step 6 – Thermal curing and conditioning. Peel the dried films and cure them at 130–140 °C for

10 min to promote ester-type crosslinking. Condition all specimens at 23 ± 2 °C and 50 ± 5% relative humidity for at least 48 h before mechanical testing.

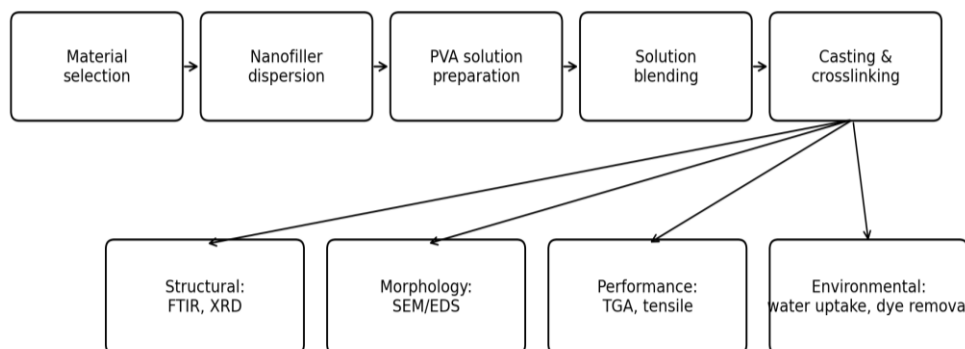


Figure 1. Proposed synthesis and characterization workflow for the polymer nanocomposite system.

3. Structural and morphological characterization

FTIR spectra are collected from 4000 to 500 cm^{-1} to identify changes in O–H stretching, C–O/C–O–C bands, carbonyl formation after crosslinking, and possible interactions between PVA, GO, and TiO_2 . XRD is recorded over $2\theta = 5\text{--}60^\circ$ to assess PVA crystallinity and to identify whether GO or TiO_2 reflections remain distinct after incorporation. A reduction or broadening of filler-related peaks is interpreted together with microscopy rather than as stand-alone proof of exfoliation.

SEM is used on cryo-fractured, sputter-coated cross sections to evaluate voids, agglomerates, crack paths, and filler distribution. EDS mapping tracks titanium spatially in P2 and P3. At least five fields of view from three specimens per formulation are analyzed. Image analysis records agglomerate area fraction and characteristic cluster size so that qualitative micrographs are supported by a quantitative dispersion metric.

4. Thermal, mechanical, and environmental performance

Thermogravimetric analysis (TGA) is performed from 30 to 600 °C under nitrogen at 10 °C min^{-1} . The temperatures at 5% mass loss (T5) and maximum degradation rate are reported. Differential scanning calorimetry may additionally be used to monitor glass-transition or melting-related changes. Tensile properties are measured on strip specimens using a constant crosshead rate; at least five replicates are tested per formulation. Tensile strength, Young's modulus, and elongation at break are calculated from engineering stress–strain curves. Water uptake is determined from dry mass (Wd) and equilibrated wet mass (Ww) using water

uptake (%) = $[(Ww - Wd)/Wd] \times 100$. Dimensional stability is also recorded because high swelling may compromise industrial use. For environmental testing, 50 mg of film is contacted with 50 mL of 20 mg L^{-1} methylene-blue solution at pH 7 under controlled agitation. Concentration is measured by UV–visible spectroscopy at the dye absorption maximum. Removal efficiency is calculated as $R(\%) = [(C_0 - C_t)/C_0] \times 100$, and adsorption capacity as $q_t = (C_0 - C_t)V/m$. Dark adsorption is measured first; where TiO_2 is present, a second test under UV-A or simulated solar irradiation may be used to separate adsorption from photocatalytic contribution.

5. Quality control and statistical analysis

All balances, pH meters, spectrophotometers, and thermal/mechanical instruments are calibrated before use. Specimen thickness is measured at five positions, and only samples within ±10% of the target thickness are compared. The primary statistical test is one-way analysis of variance (ANOVA) followed by Tukey's post-hoc comparison at $p < 0.05$. Correlation analysis is used to link agglomerate fraction, crystallinity indicator, T5, tensile strength, water uptake, and dye removal. A formulation is considered industrially promising only if environmental performance improves without a severe loss of mechanical integrity or excessive water uptake.

Results

Because the present article is a methodology paper rather than a report of completed laboratory experiments, Table 3 provides a literature-calibrated illustrative dataset. The values are included to demonstrate how the proposed measurements should be compared

and interpreted; they must not be presented as measured experimental data. The trends are consistent with prior observations that well-dispersed graphene-family fillers can improve mechanical response [7,10,14], oxide and hybrid

fillers can increase functional surface area [12,13], and environmental performance depends on both active sites and accessibility of the dispersed phase [8,11].

Table 3. Illustrative expected outcomes from the proposed experimental design

Code	T5 (°C)	Tensile strength (MPa)	Water uptake (%)	MB removal (%)	Interpretation
P0	243	31.2 ± 1.8	34.5 ± 2.1	18.4 ± 1.9	Baseline polymer; limited active surface
P1	257	40.8 ± 2.0	39.1 ± 2.4	63.7 ± 2.7	GO improves strength and adsorption
P2	266	36.9 ± 1.7	31.8 ± 1.8	77.8 ± 2.5	TiO ₂ improves thermal and functional response
P3	279	44.6 ± 2.2	33.0 ± 2.0	91.5 ± 2.2	Best multifunctional balance if dispersion is stable

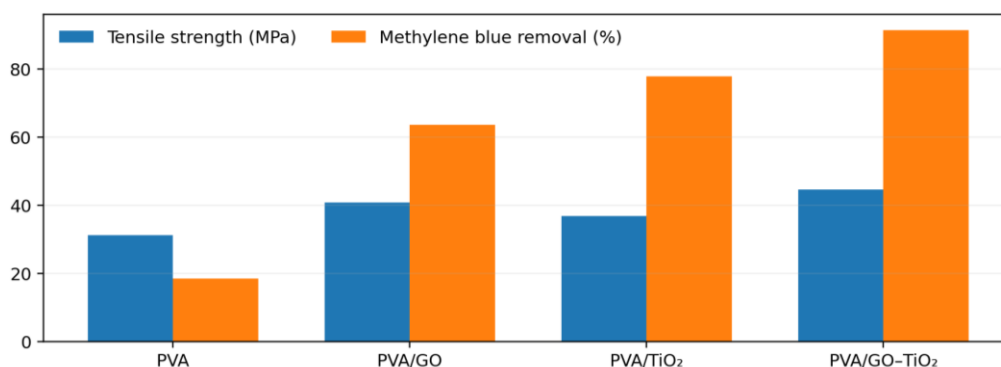


Figure 2. Illustrative tensile-strength and methylene-blue-removal comparison (not measured data).

The illustrative P1 result represents the expected effect of a low GO loading when sheets are sufficiently dispersed to create a reinforcing interphase. A rise in tensile strength with a moderate rise in water uptake is plausible because oxygen-containing GO groups can form hydrogen-bonding interactions with PVA while also increasing hydrophilicity. If SEM reveals large GO stacks, the opposite mechanical trend would be expected because agglomerates act as stress concentrators. This is why the proposed methodology treats microscopy and tensile testing as coupled measurements rather than independent characterization steps.

The P2 formulation is expected to show a stronger improvement in thermal stability and dye-removal activity than in tensile strength. TiO₂ particles can act as thermally stable dispersed domains and can contribute surface adsorption and, under suitable irradiation, photocatalytic degradation. However, the relatively low aspect ratio of spherical oxide nanoparticles generally provides less efficient stress transfer than a well-dispersed two-dimensional filler. The hybrid P3 formulation is therefore the key test of the methodology: if GO

helps distribute TiO₂ while the oxide reduces GO restacking, a synergistic morphology can emerge. The illustrative 44.6 MPa tensile strength and 91.5% dye removal represent this desired outcome.

Performance should ultimately be selected by a multi-criteria decision rather than by the maximum value of one property. A formulation with very high dye removal but excessive swelling, brittle fracture, or nanoparticle release would not be acceptable for reusable environmental components. Conversely, a mechanically excellent film with inaccessible active sites would have limited remediation value. The proposed framework therefore prioritizes a balanced property envelope consisting of dispersion stability, thermal durability, strength, controlled water uptake, and contaminant-removal efficiency.

Conclusion and Discussion

This methodology provides a compact but comprehensive route for synthesizing and evaluating multifunctional polymer nanocomposites intended for industrial and environmental applications. The proposed

PVA/GO/TiO₂ system is deliberately modular: GO represents a high-aspect-ratio carbonaceous reinforcement, TiO₂ represents an inorganic functional phase, and the GO-TiO₂ hybrid tests whether complementary fillers can create synergistic mechanical and environmental performance. The method controls solution preparation, sonication, blending, casting, crosslinking, conditioning, specimen thickness, and replication so that differences among formulations can be attributed primarily to nanofiller chemistry and dispersion. The characterization sequence is equally important. FTIR and XRD establish chemical interactions and structural changes; SEM/EDS verifies whether the desired nanoscale or fine microscale distribution has been achieved; TGA and tensile testing quantify industrial durability; and water uptake plus dye removal quantify environmental suitability. This structure follows the broader literature consensus that nanocomposite properties emerge from the interphase and processing-induced morphology rather than simply from the identity of the filler [3–6,14,15]. The environmental tests also reflect the growing use of polymer nanocomposites as reusable membranes and adsorbents [8,11,13]. The main limitation is that the numerical results presented here are illustrative rather than experimentally measured. Future work should execute the protocol with replicated batches, extend filler loading around the identified optimum, assess long-term leaching and reuse, and test real wastewater or industrial process streams containing competing ions and organic matter. Life-cycle considerations should also be included because a nanocomposite is not environmentally advantageous if its production energy, solvent use, or nanoparticle release outweighs its treatment benefit. With these extensions, the proposed methodology can serve as a practical platform for optimizing polymer nanocomposites for coatings, packaging, membranes, adsorption media, separation devices, and other multifunctional industrial components.

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