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Reframing Water Response in Glass Ionomer Cement: Gravimetric Effects of Yttrium Oxide Nanoparticle Substitution

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Abstract

The objective of this work was to evaluate water-related mass changes in a conventional glass ionomer luting cement following partial substitution of its powder with yttrium oxide nanoparticles (Y_2O_3). Forty discs (10 mm diameter; 2.5 mm thickness) were prepared using 0, 1, 3, or 5 wt% Y_2O_3 powder substitution (n = 10 per formulation). The same specimens underwent 24-hour water immersion, 24-hour drying, and seven additional days of re-immersion. Initial, wet, and post-drying masses defined net wet mass change and apparent mass loss as percentages. Overall formulation effects were evaluated by one-way ANOVA and Welch sensitivity analyses reconstructed from group summaries. Net wet mass change ranged from 0.338% to 0.716% at the first assessment and from 1.036% to 1.614% at the second. Y5 had the lowest mean apparent mass loss: 3.090 ± 0.165% and 3.422 ± 0.449%, compared with 5.556 ± 1.023% and 6.609 ± 1.122% in the control. These represented descriptive reductions of 44.4% and 48.2%. Conventional ANOVA indicated overall formulation differences for net wet mass change ($p = 0.0057$ and 0.0108) and apparent mass loss (both $p < 0.001$). Welch analyses also indicated overall differences at both assessments. The 5 wt% formulation had the lowest mean apparent mass loss while exhibiting higher mean net wet mass change than the control. The results describe short-term gravimetric behavior and do not establish improved mechanical performance or clinical durability.

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Introduction

Conventional glass ionomer cements are used in restorative dentistry and for luting indirect restorations. Their interaction with water is an important aspect of both setting and subsequent material behavior. Water is involved in the acid–base reaction and maturation of the cement; exposure to an aqueous environment can also permit material loss. Foundational reviews describe the development and chemistry of glass polyalkenoate cement systems [1-3]. Evaluating these processes therefore requires a distinction between water uptake, net mass change, and changes in mass after drying [4].

Setting is more complex than an instantaneous transition to a stable solid. Infrared studies have examined reaction products and the role of tartaric acid [5,6], while magnetic resonance investigations have addressed setting and commercial glass composition [7-9]. Work on secondary setting reactions and subsequent maturation provides a rationale for reporting the specimen conditioning history explicitly [10,11]. These investigations also show why a single post-setting mass cannot be assumed to represent a fully matured, water-independent reference state.

Modification of the powder phase provides one approach to altering cement behavior. Studies of nanoparticle-modified formulations have evaluated mechanical properties, antibacterial activity, microstructure, and ion release [12,13]. However, an effect on one property does not establish improvement across all properties relevant to a luting cement. Substitution also reduces the amount of original cement powder at a fixed total powder mass, introducing a compositional change that must be considered alongside any filler effect.

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The modification literature spans changes to the glass composition, powder geometry, and incorporation of additional phases. Studies have addressed strontium substitution, aluminosilicate glass particle size and fluorine content, and powder spheroidization [14-16]. Reviews of powder modifications and nanotechnology place these approaches within a wider dental-materials research context [17-19]. Recent reviews also distinguish chemical activation and potential bioactivity from general improvements in physical properties [20,21]. Accordingly, the research question should concern a defined material and endpoint rather than nanoparticle incorporation as a general claim of benefit.

Yttrium oxide nanoparticles have been investigated for biomedical applications, with their behavior depending on characteristics such as preparation and surface properties [22]. Their use in a dental cement requires evaluation within the specific cement system. Studies of Y_2O_3 synthesis and cytotoxicity demonstrate the importance of particle characterization [23]; reports of neuroprotective effects concern separate experimental models and cannot establish the safety of a modified luting cement [24]. A recent study examined surface microhardness following Y_2O_3 substitution at 1, 3, and 5 wt% [25]. The present investigation addresses water-related mass changes at these substitution levels, rather than treating Y_2O_3 incorporation itself as an unprecedented modification.

Gravimetric outcomes are particularly sensitive to the definition of baseline mass and to immersion, surface-blotting, and drying procedures. The difference between wet mass after immersion and initial mass reflects simultaneous uptake and loss processes. Accordingly, interpreting that difference as water sorption alone can obscure material loss during exposure. A study of nano-titania-enriched GIC provides a related gravimetric approach [26], but the endpoint definitions and exposure conditions must be stated explicitly for each experiment.

This in vitro study evaluated net wet mass change and apparent mass loss in an unmodified conventional glass ionomer luting cement and formulations prepared by partial powder substitution with 1, 3, and 5 wt% Y_2O_3. Measurements were made after the initial 24-hour immersion and after seven days of re-immersion to describe formulation-associated differences and changes across the two assessment intervals.

Material and Methods

Study design and materials

A conventional glass ionomer luting cement, Tokuyama Tokuso Ionomer GIC Luting Cement (Tokuyama Dental Corp., Japan), was used. Analytical-grade Y_2O_3 nanoparticles were obtained from US Research Nanomaterials, Inc. (Houston, USA). The particle size range was reported as 30–45 nm and purity as 99.99%. Forty specimens were allocated to four formulations, with ten specimens per formulation.

Powder modification

Y_2O_3 replaced an equivalent mass of the original cement powder. For each 1 g of total powder, the formulations contained the following proportions:

Formulation	Original GIC powder (g)	Y_2O_3 (g)	Y_2O_3 substitution (wt%)
Control	1.0	0	0
Y1	0.99	0.01	1
Y3	0.97	0.03	3
Y5	0.95	0.05	5

Table 1. Powder composition at a fixed total powder mass of 1 g.

The powders were weighed using a calibrated analytical balance; the stated weighing accuracy for formulation preparation was ±0.001 g. Modified powders were dry-mixed and subsequently blended using an amalgamator. The total powder mass was kept constant, and the powder-to-liquid proportion followed the manufacturer's instructions. No experimental measurement of nanoparticle dispersion within the set cement was undertaken.

Specimen fabrication

Disc-shaped specimens were prepared using a Teflon mold with a nominal diameter of 10 mm and thickness of 2.5 mm. Specimens in each formulation were prepared using multiple mixes. Cement was mixed according to the manufacturer's instructions and placed in the mold over a transparent matrix strip and glass slide. A second matrix strip and slide were placed above the mold, and light pressure was applied to remove excess material and produce flat surfaces. Specimens were allowed to set at room temperature for 20 minutes before removal. Each specimen was visually examined against the light for visible internal defects.

Water exposure and mass measurements

The gravimetric approach was adapted from Hussein [26], with the cement formulation, specimen thickness, exposure medium, and assessment sequence specified for the present study.

Initial specimen mass, W_o, was measured after setting, without a preliminary drying step, using an analytical balance with a stated reading accuracy of 0.0001 g. Three successive readings were obtained, and their mean was recorded. Each specimen was placed in an individually labeled, airtight glass container containing approximately 10 mL of deionized water. The immersion medium was renewed daily.

The same ten specimens per formulation underwent both assessments. After the initial 24-hour immersion, specimens underwent wet weighing and drying, then were returned to water for seven additional days before the second assessment. Before wet weighing at each assessment, specimens were lightly blotted with filter paper until no surface moisture was visible and were immediately weighed to obtain W_1. Specimens were then placed in an incubator at 37°C for a fixed 24-hour period and weighed to obtain W_2. Drying was not continued through repeated weighing cycles to a constant-mass endpoint.

Endpoint definitions

Two mass-normalized outcomes were calculated using the recorded masses:

Net wet mass change (%) = $100 \times (W_1 - W_o) / W_o$.

Apparent mass loss (%) = $100 \times (W_o - W_2) / W_o$.

Net wet mass change reflects net mass gain after concurrent uptake and loss rather than water uptake alone. This gravimetric measurement does not identify the chemical composition of material released into the immersion medium and may be influenced by drying-associated changes in the cement. Both outcomes are reported as percentages, without conversion to volume-normalized units.

Statistical analysis

Results are presented as mean ± standard deviation for ten specimens per formulation at each assessment. Descriptive relative differences in apparent mass loss between Y5 and the control were calculated as 100 × (control mean − Y5 mean) / control mean. These percentages describe differences between group means and are not measures of clinical efficacy. One-way ANOVA was reconstructed separately for each endpoint at each assessment using the reported group means and standard deviations and n = 10 per formulation. For four groups, the between-group and within-group degrees of freedom were 3 and 36, respectively. Between-group sums of squares were calculated as $\Sigma\, n(m_i - \bar{m})^2$, and within-group sums of squares as $\Sigma(n-1)s_i^2$. F statistics were obtained from the corresponding mean squares and evaluated against the F distribution. All ten specimens contributed to each summary, and dispersion values are standard deviations, as confirmed by the authors. The calculation uses rounded group summaries. A

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significance threshold of 0.05 was applied to the four omnibus tests, with no adjustment for multiplicity; exact p values are provided. Welch one-way ANOVA was additionally reconstructed from the same summaries as a sensitivity analysis allowing unequal group variances. These calculations do not verify within-group normality or independence. The Welch analysis does not address dependence among specimens prepared from a common mix. Paired temporal effects and concentration-by-assessment interactions were not estimated from the marginal summaries. Individual post hoc contrasts were not included in the present analysis.

Results

Net wet mass change

All formulations had positive mean net wet mass change at both assessment intervals (Table 2). At 24 hours, means were 0.338% for the control, 0.556% for Y1, 0.529% for Y3, and 0.716% for Y5. Y5 therefore had the largest mean value, although the ordering of Y1 and Y3 did not indicate a strictly monotonic concentration relationship.

At the second assessment, mean values were higher than their corresponding 24-hour values in every formulation. Y3 had the highest mean, 1.614%, followed by Y5 at 1.591%, Y1 at 1.222%, and the control at 1.036%. In the reconstructed conventional ANOVA, the overall between-formulation test for net wet mass change gave $F(3,36) = 4.926$, $p = 0.0057$ at the first assessment and $F(3,36) = 4.302$, $p = 0.0108$ at the second assessment. These omnibus tests do not identify significant individual formulation contrasts or establish a temporal effect.

Apparent mass loss

Y5 had the lowest mean apparent mass loss at both intervals. At 24 hours, the mean was 3.090%, compared with 5.556% for the control, 5.853% for Y1, and 4.287% for Y3. At the second assessment, the corresponding values were 3.422%, 6.609%, 7.656%, and 6.653%.

Relative to the control, Y5 had mean apparent mass loss that was 2.466 percentage points lower at 24 hours and 3.187 percentage points lower at the second assessment. The descriptive relative reductions were 44.4% and 48.2%, respectively. Y1 had higher mean values than the control at both intervals. Y3 was lower than the control at 24 hours but had a similar mean at the second assessment. Apparent mass loss was numerically higher at the second assessment than at 24 hours in every formulation. Reconstructed overall between-formulation tests gave $F(3,36) = 16.321$ at the first assessment and $F(3,36) = 17.468$ at the second assessment (both $p < 0.001$). These results do not establish which individual contrasts are significant.

Values are mean ± standard deviation; n = 10 per formulation at each assessment. Both endpoints are expressed as percentages of initial post-setting mass. No pairwise or temporal significance symbols are assigned.

Tests compare formulations within each assessment and were reconstructed from rounded means and standard deviations. P values are unadjusted omnibus results; they do not identify individual significant contrasts or temporal effects. No normality or independence assessment is implied by these calculations.

Welch tests allow unequal group variances. The calculations use the same n = 10 and SD summaries as Table 3. They do not test within-specimen temporal effects or account for possible dependence within preparation mixes.

Discussion

Conventional and Welch omnibus analyses indicated overall formulation differences in net wet mass change and apparent mass loss at both assessments. Agreement between these analyses supports the overall finding when unequal group variances are considered, although neither establishes which individual formulations differ or whether a concentration-response relationship exists.

The main descriptive observation was the lower apparent mass loss in the 5 wt% Y_2O_3 formulation at both assessment intervals. This occurred alongside a higher net wet mass change than in the control. The two outcomes should be considered together: lower mass loss after drying does not necessarily imply lower water uptake, and a positive wet mass difference does not isolate the amount of water absorbed.

Interpretation of the gravimetric endpoints

The choice of endpoint matters for the interpretation of this study. $W_1 - W_o$ is a net quantity influenced by water entering the specimen and material leaving it. If material loss differs among formulations, differences in this quantity cannot be attributed exclusively to water uptake. Similarly, $W_o - W_2$ represents a mass difference after a specified drying procedure rather than direct chemical measurement of dissolved constituents. Water involved in cement maturation further complicates interpretation of a post-drying mass [4].

These considerations support using operational endpoint names and reporting the equations alongside the results. They also preclude direct comparison of the percentage values with published measurements in $\mu g/mm^3$ without the necessary mass and volume information. No conformity with an ISO acceptance threshold is inferred from the present gravimetric measurements.

Possible effects of powder substitution

The lower apparent mass loss in Y5 is compatible with a change in the amount or accessibility of material susceptible to loss during water exposure. However, the experiment does not distinguish physical filling, altered reaction chemistry, changes in porosity, or differences in drying behavior. Substitution also decreases the mass of original reactive cement powder while holding the total powder-to-liquid proportion constant. Consequently, attributing the result solely to nanoparticle reinforcement would exceed the evidence.

The data do not describe a uniform concentration response. Y1 had higher mean apparent mass loss than the control at both intervals, and Y3 had a mean like the control at the second assessment. The relatively low Y5 values therefore warrant targeted investigation rather than a claim that every increase in Y_2O_3 concentration improves performance. Four tested levels are insufficient to establish an optimum, especially when relevant functional and safety outcomes remain unmeasured.

Studies of nanoparticle-modified GICs provide a broader context for evaluating formulation-dependent behavior [12,13]. A study of nanotitania-enriched cement also examined the influence of storage medium and time [26]. These studies support treating exposure conditions as part of the experimental question; they do not validate a specific mechanism for the present Y_2O_3 formulation. The recent Y_2O_3 microhardness study concerns a different measured property [25]. Its findings should not be substituted for mechanical testing of the specimens or formulation batches assessed here.

Comparison with other modified cement systems

Hydroxyapatite and fluoroapatite have been incorporated into conventional GICs, with studies examining mechanical behavior, bonding, and fluoride release [27,28]. Other investigations have combined hydroxyapatite with zirconia or silica and have considered porous spherical hydroxyapatite [29-31]. These are relevant formulation comparisons, but their additives, reaction chemistry, and outcomes differ from the present Y_2O_3 substitution. A finding in an apatite-containing material therefore does not establish an equivalent mechanism here.

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Nanoclay modification has been evaluated across strength, stiffness, and setting outcomes [32]. Bioactive-glass additions have been studied in relation to physicochemical properties, biocompatibility, and antimicrobial behavior [33,34,35], while reactive glass fibers represent another route to changing fracture resistance [36]. Collectively, this literature supports testing multiple properties of a candidate formulation; mass change alone cannot establish reinforcement, antibacterial action, or bioactivity.

Evidence from resin-modified GICs requires caution. Research has examined competition between their setting reactions, water-related physical changes, ion release, and biocompatibility [37-40]. Investigations of nanoapatite modifications and particle-size or powder-to-liquid changes also concern resin-modified materials [41-43]. Their polymerization component makes direct transfer of results to the conventional acid–base luting cement used here inappropriate. Similarly, surface-coating studies concern a separate intervention that can influence measured physical properties [44].

Recent nanoparticle studies reinforce the need for endpoint-specific comparisons. A 2025 investigation of zirconium oxide nanoparticles examined mechanical properties together with water sorption and solubility [45]. A 2026 study of green-synthesized titanium dioxide nanoparticles combined physical testing with preliminary computational safety screening [46]. Neither evaluates this Y_2O_3 formulation. A study of chlorhexidine-loaded mesoporous silica illustrates a further distinct strategy involving an antimicrobial payload [47]. Such work provides context for future testing without demonstrating antimicrobial or safety benefits for the present cement.

Assessment interval and experimental interpretation

Both mean net wet mass change and mean apparent mass loss were higher at the second assessment than at 24 hours in all groups. Moreover, two intervals do not establish equilibrium hydration, a diffusion coefficient, or a long-term degradation rate.

The same specimens were dried after the first assessment and then re-immersed for seven additional days. The second assessment therefore reflects a prior immersion–drying cycle followed by re-immersion, rather than seven days of uninterrupted immersion from baseline. Time-associated differences cannot isolate exposure duration from the effects of the intervening drying step. The initial mass was recorded after setting, and subsequent drying was limited to 24 hours without establishing constant mass. Apparent mass loss may consequently include changes in water content as well as loss of cement constituents.

Exposure medium is another source of non-equivalence. Earlier studies have examined fluoride release into deionized water and artificial saliva, early aluminum release during water exposure, and longer interaction with lactic acid [48-50]. Research has also addressed saliva-associated surface hardness and the effects of pH on mechanical properties and ion release [51,52]. These studies justify considering aqueous chemistry and exposure duration during interpretation, but the present experiment cannot predict behavior in saliva or under acidic challenges. Moreover, formulations combining polyacid changes with apatite additives introduce simultaneous chemical modifications that must be distinguished from powder substitution alone [53].

Relevance to luting applications

For a luting cement, resistance to aqueous material loss is a potentially relevant property, but clinical performance also depends on setting behavior, film thickness, retention, and mechanical behavior under service conditions. None of those outcomes was measured in this experiment. The observed lower mean apparent mass loss cannot therefore establish improved marginal integrity, reduced recurrent caries, or longer restoration survival.

Likewise, a higher net wet mass change cannot be described as beneficial dimensional compensation without dimensional measurements. The findings identify Y5 as a formulation for further evaluation, not as a formulation ready for clinical recommendation.

Limitations and further investigation

The study examined one commercial luting cement, three substitution levels, one immersion medium, and two short-term assessment intervals. Nanoparticle distribution, phase identity within the modified material, and interactions with the cement matrix were not experimentally characterized. Mechanical properties, dimensional changes, fluoride or yttrium release, and biological responses were not measured. These boundaries restrict both mechanistic interpretation and extrapolation to other products or oral conditions. The analyses were reconstructed from rounded group summaries, and the four omnibus tests were not adjusted for multiplicity. Welch analyses address unequal variances but do not establish within-group normality or specimen independence. Multiple mixes were used, but the number of mixes and their specimen allocation were not specified; possible mixing-related dependence limits inference. No paired temporal tests or individual formulation contrasts were evaluated. Further investigation should first establish reproducible preparation and gravimetric procedures across independently prepared batches. Characterization of particle dispersion and set-cement structure would help distinguish competing explanations for the mass changes. Functional testing relevant to luting applications, longer exposure periods, and appropriate biological evaluation would then clarify whether the lower apparent mass loss occurs without unfavorable changes in other properties.

Conclusion

Under the reported experimental conditions, the 5 wt% Y_2O_3-substituted glass ionomer luting cement had the lowest mean apparent mass loss after the initial 24-hour immersion and after seven days of re-immersion, while its mean net wet mass change exceeded that of the unmodified cement. The results describe short-term gravimetric behavior and do not establish improved strength, clinical durability, or an optimal substitution level. Conventional and Welch analyses reconstructed from the group summaries indicated overall formulation differences at both assessments. Individual contrasts, temporal effects, and the material mechanisms underlying these observations require further evaluation.

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Table 2. Net wet mass change and apparent mass loss after water exposure.

Formulation	Net wet mass change at 24 h (%)	Net wet mass change after 7 d re-immersion (%)	Apparent mass loss at 24 h (%)	Apparent mass loss after 7 d re-immersion (%)
Control	0.338 ± 0.162	1.036 ± 0.176	5.556 ± 1.023	6.609 ± 1.122
Y1	0.556 ± 0.356	1.222 ± 0.518	5.853 ± 0.666	7.656 ± 2.469
Y3	0.529 ± 0.2	1.614 ± 0.406	4.287 ± 1.557	6.653 ± 0.443
Y5	0.716 ± 0.045	1.591 ± 0.534	3.090 ± 0.165	3.422 ± 0.449

Table 3. Conventional one-way ANOVA reconstructed from group summaries with n = 10 per formulation.

Endpoint	Assessment	F(3,36)	Unadjusted p
Net wet mass change	Initial 24-hour immersion	4.926	0.005725
Net wet mass change	Seven-day re-immersion	4.302	0.010804
Apparent mass loss	Initial 24-hour immersion	16.321	7.32×10^{-7}
Apparent mass loss	Seven-day re-immersion	17.468	3.63×10^{-7}

Table 4. Welch sensitivity analysis reconstructed from group summaries.

Endpoint	Assessment	F	Numerator df	Denominator df	Unadjusted p
Net wet mass change	Initial 24-hour immersion	17.83	3	16.239	2.18e-05
Net wet mass change	Seven-day re-immersion	7.464	3	17.773	0.00194
Apparent mass loss	Initial 24-hour immersion	66.334	3	15.888	3.32e-09
Apparent mass loss	Seven-day re-immersion	90.206	3	18.578	2.93e-11