

# Bracket Bond Strength Effects of Incorporation of NovaMin into an Orthodontic Bonding Resin

Jeffrey Waterhouse  
*Marquette University*

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BRACKET BOND STRENGTH EFFECTS OF INCORPORATION OF  
NOVAMIN INTO AN ORTHODONTIC BONDING RESIN

by

Jeffrey D. Waterhouse, D.D.S.

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Marquette University,  
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ABSTRACT  
BRACKET BOND STRENGTH EFFECTS OF INCORPORATION OF  
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Jeffrey D. Waterhouse, D.D.S.

Marquette University

Despite advances in caries research and the development of protective mechanisms and materials, orthodontic white spot decalcification remains a significant problem. Increased plaque buildup around orthodontic appliances, combined with inadequate hygiene and a susceptible patient, can lead to unesthetic demineralized lesions in enamel.

Relatively new to the arena of caries prevention, NovaMin, a bioactive glass, has shown some promise in areas of remineralization of early lesions, protection against acidic challenge, and has demonstrated antiplaque properties. This study explored a new application of the material, incorporation into an orthodontic bonding composite resin. Objectives of the study were: to determine if the experimental resin could release ionic precursors to enamel formation in solution, and the effects on bracket shear bond strength.

For ion release, NovaMin concentrations of 7.5, 15 and 22.5 wt% were incorporated into a commercially available resin, TransBond LV. Resin discs were immersed in 5 mL of both deionized water and lactic acid (pH~5). Calcium ion concentrations were measured using a calcium-selective electrode. Solutions were replaced every 24 hours for the first week, then remained unchanged for an additional 5 weeks. Controls were the resin without the modification and solutions without a resin disc. Concentrations declined rapidly over the first two days, reaching levels below that of the control solutions. Without solution replacement, a significant increase in calcium ion concentration was observed for NovaMin levels of 15 and 22.5%.

For bond strength, 39 extracted human premolar teeth were tested in three groups of 13. Standard twin brackets were bonded with either TransBond LV or the same resin with 15 or 22.5% NovaMin incorporated. Shear bond strength was tested using a universal testing machine. ARI scores were also obtained. There was no significant difference in mean shear bond strength between the three groups (TransBond LV ( $15.13 \pm 4.18$ MPa), TransBond LV + 15% NovaMin ( $13.55 \pm 2.84$ MPa), and TransBond LV + 22.5% NovaMin ( $13.27 \pm 4.34$ MPa)). There was, however, a significant difference in ARI scores between the group without the modification and both NovaMin groups, with the as-provided TransBond LV showing less residual adhesive on the enamel after debond.

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CHAPTER I  
INTRODUCTION



Placement of fixed orthodontic appliances during comprehensive treatment increases the available surface area for plaque retention in patients and makes effective oral hygiene more difficult. The potential exists for cariogenic bacteria to demineralize the enamel of these patients, compromising the esthetic outcome of treatment through the appearance of white spot lesions. Prevalence of these lesions varies widely in reported literature from 2-96% (Chang et al. 1997). Much research has been undertaken to develop methods to eliminate, prevent or improve the appearance of these lesions.

The purpose of this study was to explore the potential use of a relatively new product, NovaMin, in the prevention of white spot demineralization. Recently, claims have been made by the manufacturer that NovaMin has the capability to release essential ionic precursors of enamel upon exposure to the oral environment (Burwell et al. 2009). These minerals theoretically repair damaged and demineralized enamel, preventing and even reversing the initial caries process.

This study investigated the effects of incorporation of NovaMin into a commercially available orthodontic bonding resin. Currently, no studies have been published utilizing NovaMin in this manner. Calcium ion release profiles were obtained, varying the weight percentage of incorporated NovaMin. The effects on bracket shear bond strength in vitro were also sought.

CHAPTER II  
REVIEW OF THE LITERATURE

## **Enamel Decalcification**

### *Etiology*

White spot enamel decalcification begins with the accumulation of cariogenic bacteria. These bacteria, through the metabolism of sucrose provided by the host's diet, produce lactic acid that begins drawing minerals from the enamel surface. The surface is lost to plaque fluid and saliva, then subsurface minerals reconstitute the surface, continuing the process. Through repeated episodes of mineral loss, the enamel increases in porosity which changes the optical properties and creates a chalky, whitish appearance. This process may continue until no subsurface minerals are left, at which point frank cavitation occurs (Chang et al. 1997).

Orthodontic fixed appliances provide plaque protection from physical forces that naturally clean tooth surfaces. Movement of food and oral musculature are reduced, as well as access to saliva flow that contains buffers and remineralizing ions. Plaque itself is also a physical barrier that limits acid diffusion from the tooth surface and prevents the penetration of necessary salivary ions (Chang et al. 1997).

The process may also arrest or reverse under appropriate conditions. Salivary minerals, fluoride, or therapeutic intervention may aid the remineralization process. Examples have been shown in the literature of orthodontic patients with considerable white spot lesions spontaneously resolving with no intervention (Chang et al. 1997).

### *Prevalence*

One classically referenced study (Gorelick et al. 1982) reported the prevalence of white spot lesions in a private orthodontic office to be present in 50% of all patients and 10.8% of all teeth treated. Highest incidence was on maxillary laterals and lowest was in the maxillary posteriors. No lesions were found on the lingual of bonded canine to canine retainers, indicating that access to salivary flow is important. No differences were found with regard to treatment

time. The authors of the study developed a visual scale system to describe the severity of the white spot lesions, one that has been adopted in modified versions in many subsequent studies.

Other studies have confirmed these findings. In a comparison of two orthodontic offices (Artun and Brobakken 1986), one more closely monitored for oral hygiene and adherence to a fluoride rinse regimen, incidence was higher on maxillary laterals and mandibular canines and premolars. The group more closely monitored had white spot scores similar to a control group which had no treatment, but the average follow-up after debond was relatively longer. A more recent study (Lovrov et al. 2007) correlated white spot lesions to oral health parameters commonly used in periodontal therapy. They found 24.9% of teeth formed new lesions or increased in number of lesions. More were found in premolars (34.4%) and anteriors (28.1%) than molars (11.8%). A positive correlation was found between the lesions and gingival attachment level, oral hygiene and fluoride use.

Although white spot lesions can lessen over time, they can still be a significant problem years after treatment. In a study (Ogaard 1989) of treated versus untreated 19 year old patients, 4% of treated patients had no white spots after treatment. The control group had 15% without white spots even with no treatment. The author stated that lesions in the presence of fluoride likely seal with a surface layer of fluorapatite, preventing diffusion of minerals from saliva to subsurface layers and delaying recovery. Average time post treatment was 5.7 years.

Lesions have also been shown to form rapidly, extending to 75  $\mu\text{m}$  with 25% enamel loss after as little as 4 weeks (Glatz and Featherstone 1985). Another study came to the same conclusion, with measurable demineralization found in extracted premolars after one month, with 15% mineral loss and 50  $\mu\text{m}$  depth (O'Reilly and Featherstone 1987). This is clearly a problem with orthodontic therapy, as treatment times can last several years.

Orthodontists are aware of the problem. A survey provided to orthodontists in the Netherlands illustrates this point (Derks et al. 2007). The questionnaire, which was returned by

78% of orthodontists, reported that 68% considered it necessary to develop practice guidelines for prevention of white spot lesions, 95% always give oral hygiene instruction at the start of treatment, and 99% take extra measures if demineralization is detected. Awareness of scientific evidence is problematic, however, as high fluoride toothpaste and chlorhexidine is rarely prescribed. Fluoride rinse is prescribed most often (51.5% always do), and 56% use a fluoride releasing bonding material. Oral hygiene is seen as the major cause, as 62% of respondents said they had patients with inadequate hygiene.

Lesions can be managed post-treatment. One method that has been studied involves microabrasion. Visible demineralization can be reduced by an average of 83% with the use of 18% HCl and pumice solution (Murphy et al. 2007). Digital photographs taken before and after therapy can be used to quantify the changes through the use of image processing software, which has been shown to be a reproducible and reliable method (Livas et al. 2008).

### *Prevention*

Considerable research effort has been spent on the prevention of orthodontic white spot lesions. Methods include the use of fluoride in various delivery methods, daily chlorhexidine rinses, application of enamel sealants, and bonding materials. A systematic review covering research from 1970 to mid 2002 stated that fluorides and chlorhexidine had an inhibiting tendency on demineralization (Derks et al. 2004). Sealants had almost no inhibiting effect. Fluoride releasing bonding materials had no statistically significant effect. Each of these methods will be reviewed further.

Fluoride use has been the most consistently utilized, though which method or combination of methods is most effective is still unknown due to a lack of quality scientific evidence. Based on a Cochrane review, there is some evidence for the use of a daily 0.05% sodium fluoride rinse as well as the use of a fluoride releasing glass ionomer bonding cement

instead of composite resin (Benson et al. 2004). Another systematic review published recently concluded that there is a reduced incidence of decalcification with topical fluorides in addition to fluoride toothpaste, but there is no evidence for a superior method (Chadwick et al. 2005). It was suggested that potency of the fluoride might be important.

One of the basic problems with most fluoride delivery methods is the necessity for adequate patient compliance. Since most lesions occur in patients with inadequate oral hygiene, methods have been developed that do not require active patient participation. One method is fluoride varnish application. In a study (Farhadian et al. 2008) of patients who required premolar extractions as part of comprehensive therapy, a split-mouth investigation was carried out in which one premolar received a fluoride varnish while the contralateral premolar served as a control. A 40% reduction in lesion depth was observed after extraction. An *in vitro* study also demonstrated a protective effect of fluoride varnishes, showing a 38% reduction in lesion depth on bovine incisors treated with a varnish and subjected to 35 days of cariogenic cycling (Demito et al. 2004). These studies, however, were carried out over a relatively short time interval compared to comprehensive therapy.

Fluoride-releasing elastomeric modules have also been studied. Theoretically, a module would provide a reservoir of fluoride that is replenished at every adjustment appointment and is not reliant upon patient cooperation. In a randomized, controlled clinical trial, a split-mouth design was used to test these elastomers (Mattick et al. 2001). Patients were followed throughout the entire course of treatment, and they were encouraged to use a fluoride rinse daily. Significantly more enamel defects were found in the control teeth, and defects were more severe.

The cariostatic mechanism of fluoride is complex. It involves the formation of calcium fluoride-like globules on the surface of enamel. These globules also contain traces of phosphate. They can persist for weeks and months and are fairly insoluble at neutral pH. At low pH, fluoride and calcium ions are released, stabilizing enamel and enhancing remineralization. Below pH 4.5,

however, the solubility limit of fluorapatite is exceeded, and the cariostatic potential of fluoride is lost. Bacteria associated with caries may lower pH in plaque below this level (Ogaard 2001).

It has been shown that bacteria adhere to orthodontic materials (Lim et al. 2008). A study (Demling et al. 2010) was conducted to determine if bacterial adhesion to brackets could be reduced by coating with PTFE (polytetrafluoroethylene; Teflon). Primary molars were bonded in a split-mouth design on 13 patients for 8 weeks. It was found that  $4.0 \pm 3.6\%$  of the treated surface was covered with biofilm versus  $22.2 \pm 5.4\%$  of uncoated controls. Longevity of the coating was in question, however.

Sealants were developed to provide a physical barrier to plaque acids and demineralization. Many studies have been carried out evaluating the effectiveness of these products with varying results. Most studies have been carried out in vitro utilizing extracted teeth and demineralizing solutions. As early as the 1970's, two of these laboratory studies showed the preventative effect of sealing teeth, where treated teeth showed less decalcification (Hughes et al. 1979, Tillery et al. 1976).

Sealant integrity is a factor in the protective effect. Another in vitro study showed that 80% of sealed teeth showed no demineralization, but lesions were present where the protective layer was discontinuous (Frazier et al. 1996). A later study confirmed this finding (Tanna et al. 2009). Sealants with more resistance to abrasion have been developed by adding fillers. Laboratory tests of these materials have shown protective effects to the enamel up to 97% compared to controls even after simulated toothbrush abrasion (Hu and Featherstone 2005, Buren et al. 2008). These studies also showed the filled sealant to be more effective than an unfilled sealant or fluoride varnish.

Few successful clinical studies have been carried out on the use of sealants. One recent study (Benham et al. 2009) utilized a split mouth design with 60 patients, placing sealant on anterior teeth gingival to the bracket. Sealants were placed from 2 weeks to 3 months after

bonding and were removed after 15-18 months. Visual lesions were detected on 6 treated teeth versus 22 untreated teeth. Different brackets and bonding techniques were used, however, complicating the interpretation of the results. Another recent study (Ghiz et al. 2009) showed similar results with the same study design comparing a sealant with a self-etching primer. Decalcification scores were reported for 27.5% of the self-etch primer group compared with 13.9% in the sealant group. Hygiene compliance was also found to be significant. The lack of protection of self-etching primers was previously found in the *in vitro* counterpart to this study, where 100% of teeth treated with the primer displayed decalcified lesions compared to 50% of sealed teeth (Tanna et al. 2009). Another attempt at protection involved the incorporation of an antibacterial monomer into a self-etching primer to reduce bacterial adhesion. A randomized controlled trial showed no benefit with respect to plaque accumulation or demineralization over 12 months of observation (Paschos et al. 2009).

Another method of white spot lesion prevention has been in the development of ion-leachable bonding agents. The known protective effect of fluoride led to the development of fluoride-releasing adhesives. In the mid-1990's, a resin-modified glass ionomer adhesive, Fuji Ortho LC, was introduced and was claimed to have superior decalcification prevention and bond strength equal to that of conventional resins. An initial clinical study of the material claimed no decalcification present on any teeth upon debond and a bonding success rate of 96.8% in 150 full arch cases, strong claims that have not since been substantiated (Silverman et al. 1995). However, there is evidence to show the potential benefit of this material. More recent *in vitro* studies showed significantly smaller lesion depth and mineral loss compared to other fluoride-releasing bonding materials and sealants (Paschos et al. 2009, Sudjalim et al. 2007). A potential drawback to this material is higher levels of bacterial adhesion, suggesting careful removal of all adhesive after bracket placement is necessary (Lim et al. 2008).



Fluoride-releasing bonding materials have been shown to release sustained, low levels of fluoride over time. A study (McNeill et al. 2001) tested several available materials for release using an ion-selective electrode. An initial high rate of release was shown to decrease after just a few days, but lower levels were detectable for long periods of time at a concentration shown previously to be effective for protection.

A shift in caries research has prompted the development of materials that can remineralize tooth structure. Fluoride, in the presence of calcium and phosphate ions, promotes formation of fluorapatite. This process is reported to be calcium and phosphate limited due to the molecular structure of fluorapatite, requiring 10 calcium ions and 6 phosphate ions for every 2 fluoride ions (Reynolds 2008). Several materials have been developed that claim to release these necessary calcium and phosphate ions for remineralization to occur.

Amorphous calcium phosphate (ACP), usually in the form of calcium sulfate and ammonium phosphate, is supplied in a dual chamber device that mixes upon application and forms ACP intraorally. In an unstabilized form, ACP rapidly precipitates and becomes inactive. The precipitates may promote calculus formation and sequester fluoride in the form of fluorapatite unbound to enamel (Reynolds 2008).

Forms of stabilized ACP have been developed. Zirconium stabilized particles (Zr-ACP) have been shown to prevent rapid precipitation (Skrtic et al. 2002). Much research has been done involving incorporation of Zr-ACP into resin composites. Initial testing showed the resins had low strength, but modifications have been made to improve mechanical properties by milling particles to a smaller diameter, increasing dispersion in the resin matrix (Lee et al. 2007). Calcium and phosphate ion release was measured by immersing resin discs in saline and continuously stirring up to 22 days. The maximum concentration of calcium ions was measured at 0.7-1.0 mmol/L. In a follow-up study (O'Donnell et al. 2008), the discs were immersed for up to 6 months, with calcium ion concentration reaching 1.2 mmol/L. Ion levels were reported to be

above the minimum level needed for mineral re-deposition to occur in enamel. Another in vitro study (Langhorst et al. 2009) of the ACP composite material tested resistance to demineralization. Artificial enamel caries lesions were created in enamel specimens and covered with 1mm thick layers of 40% ACP composite, a fluoride-releasing cement, or nothing as control. The specimens were subjected to 1 month of cyclical demineralizing and remineralizing solutions with continuous stirring. The ACP composite showed a 14% gain in mineral content compared to 4% gain with the fluoride-releasing cement and 55% loss in the control.

One commercially available ACP resin, Aegis Ortho, has been tested in vitro for demineralization protection. It was found to be better than the control resin but similar to a resin-modified glass ionomer (Uysal et al. 2009).

Another stabilized form of ACP comes in the form of casein phosphopeptide stabilized ACP (CPP-ACP; Recaldent). It is claimed to bind ions in high concentrations, adhering to both pellicle and plaque with the ability to diffuse ions into subsurface lesions. It has been shown to have anticariogenic activity in laboratory, animal and human experiments as well as in one randomized controlled trial (Reynolds 2008). One report showed a topical cream applied to existing white spot lesions twice a day after brushing improved regression of the lesions 31% over controls (Bailey et al. 2009). Another study (Mazzaoui et al. 2003) investigated the effects of incorporating CPP-ACP into a glass-ionomer cement. At a level of 1.56%, the CPP-ACP was reported to increase microtensile bond strength to dentin by 33% and compressive strength 23%. An enhanced protection of dentin to cariogenic challenge in vitro was reported. Calcium, phosphate and fluoride ion release from discs was measured. Calcium release was low and only at low pH, phosphate was high at both neutral and acidic pH, and fluoride was higher compared to control values at neutral and acidic pH.

Calcium sodium phosphosilicate, a bioactive glass, has recently become available as a remineralizing alternative. Bioactive glass (Bioglass) was developed in 1969 for orthopedic

applications. It is capable of bonding to bone and soft tissue and has gene activation properties. Bioglass has been used since the mid 1980's for middle ear prostheses and endosseous alveolar ridge maintenance implants. Its limited mechanical strength and low toughness prevent its use in load bearing applications (Hench 2006).

When Bioglass is inserted into living tissues, the first reaction stages release soluble ionic species and create a bi-layer of hydrated silica and hydroxycarbonate apatite on the surface of the glass (Hench 2006). NovaMin was developed to take advantage of this process. A derivation of the original Bioglass formulation, NovaMin has a composition of 45% SiO<sub>2</sub>, 24.5% CaO, 24.5% Na<sub>2</sub>O and 6% P<sub>2</sub>O<sub>5</sub> with an average particle size of ~2μm (Cerruti et al. 2005). Exposure to the oral environment releases sodium ions from the structure through an exchange with hydrogen ions, which in turn increases the pH of the solution and deposits a calcium phosphate layer on tooth surfaces. This layer crystallizes to form hydroxycarbonate apatite, repairing surface lesions and increasing surface hardness (Burwell et al. 2009).

The original purpose of NovaMin was for the treatment of hypersensitivity by occluding dentinal tubules and has been FDA approved for this purpose (Litkowski et al. 1997, Wefel et al. 2009). Most of the research on its remineralization capability has been internal studies published by company researchers. In vitro testing has shown an increase in microhardness when applied to artificial white spot lesions (Burwell et al. 2008). Ion release profiles have been reported to be more sustained with NovaMin-containing products than CPP-ACP products currently available (Burwell and Muscle 2009).

Other studies have shown additional potential benefits of NovaMin. An antibacterial effect was demonstrated on oral bacteria, including *S. Mutans*, possibly by the alkaline nature of the surface reactions (Allen et al. 2001). One randomized controlled trial conducted on a NovaMin-containing dentifrice showed an anti-gingivitis and anti-plaque effect with a 58.8%

reduction in gingival bleeding and 16.4% reduction in plaque growth in subjects compared to controls (Tai et al. 2006).

Independent studies on NovaMin's remineralization potential have been relatively unfavorable. An in vitro study comparing a NovaMin dentifrice to a CPP-ACP product and a regular dentifrice showed that all three protected against mineral loss in an artificial caries model to the same extent (Rehder Neto et al. 2009). Another in vitro study reported enamel lesions were hardened to a greater extent with fluoride alone than with either NovaMin or CPP-ACP products (Smith et al. 2008).

### **Bond Strength**

Many research studies have been conducted evaluating the effects on bracket bond strength of white spot lesion preventative products. A Cochrane review (Mandall et al. 2003) summarized the current literature of orthodontic bonding agents with respect to reliability and decalcification prevention. The overall evidence was weak and no conclusions could be drawn. Some of the research will be explored further with the inclusion of more recent publications.

An in vitro study of a filled resin sealant (ProSeal) showed bond strengths used with various resin composites were slightly lower than controls, but were greater than 10 MPa (Lowder et al. 2008).

Early claims of resin-modified glass ionomers were that bond strengths and failure rates were equal to resin composites, but studies since have shown otherwise. A prospective clinical trial compared Fuji Ortho glass ionomer to a conventional resin, Light Bond, in a split mouth design (Gaworski et al. 1999). Patients were followed for 12 to 14 months. The failure rate of the glass ionomer was 24.8% compared to 7.4% for the resin. Decalcification rates were similar between the two groups. Since this study, the manufacturer has suggested preparation of the enamel with 10% polyacrylic acid before bonding.

Another glass ionomer had similar results (Wright et al. 1996). Geristore was compared to a composite resin, Phase II, in a split mouth design and was found to have higher bond failure overall (8.9% versus 3.1%). No significant difference was found on anterior teeth. Bond failures were followed up to one year.

Rely-a-Bond, a fluoride releasing composite resin, was tested in a split-mouth clinical trial (Banks et al. 1997). Patients were followed for the entire course of treatment. No significant differences were found with respect to bond failures or rates of decalcification. More recently, several new fluoride-releasing adhesives were tested in vitro versus controls and were also found to have acceptable bond strengths similar to controls (Pseiner et al. 2010).

A fluoride-releasing self-etching primer, TransBond Plus, was compared to another such primer, Clearfil Protect Bond, in a randomized controlled trial in a split-mouth design (Paschos et al. 2009). Clearfil Protect Bond also contains an antibacterial monomer. Over 12 months of observation of 480 brackets, there were 5 failures with TransBond and 21 with Clearfil Protect Bond, a statistically significant difference. A complicating factor, however, was that the enamel was not separately etched for the Clearfil Protect Bond despite manufacturer's recommendations.

Aegis Ortho, a commercially available ACP-containing composite, has been shown in independent in vitro studies to have significantly lower bond strength than controls, but at a level reported to be clinically acceptable according to Foster et al. (2008). Two studies tested metal brackets (Foster et al. 2008, Dunn 2007) and one tested ceramic brackets (Uysal et al. 2010). Excess composite was not removed from Aegis-bonded brackets, a manufacturer recommendation.

CHAPTER III  
MATERIALS AND METHODS

### **Calcium Ion Release**

NovaMin (NovaMin Technology Inc., Alachua, FL) particulate powder was incorporated into a commercially available flowable composite resin, TransBond LV (3M Unitek, St. Paul, MN). NovaMin concentrations tested were 0, 7.5, 15 and 22.5 by weight percent. These percentages were decided on through a trial mix of up to 25 weight percent, where the resin reached a limit of powder incorporation while still remaining workable. Weight measurements were made using a scientific analytical balance (AG245; Mettler-Toledo, Inc., Columbus, OH). NovaMin powder was slowly incorporated into the resin by hand on a mixing pad until the mixture was homogeneous. The resin was immediately molded into discs under low light to prevent initial curing.

Resin discs (1.6 mm height by 8 mm diameter) were formed using a Teflon mold. The mold was placed on a glass slab covered with a mylar strip. The resin was expressed into the mold, covered by another mylar strip and pressed firmly with a glass slide. The discs were then light cured for 40 seconds using a standard halogen dental light curing unit (Optilux 501, SDS Kerr, CT).

The discs were recovered from the mold and excess flash removed. After 24 hours, individual discs were immersed in 5 mL solutions of either deionized water or a lactic acid solution at pH~5 contained in plastic centrifuge tubes. An example of this is shown in Figure 1. These solutions were used based on a previous study (Mazzaoui et al. 2003). Each concentration of incorporated NovaMin was tested in each solution with five identical discs made from the same mixing batch for consistency and reliability. The TransBond LV + 0% NovaMin group served as a control to assess the amount of calcium released, if any, from the resin itself. Additionally, five tubes were filled with either deionized water or lactic acid alone as controls to determine the amount of calcium present in the solutions without the experimental discs.

**Figure 1.** Resin disc immersed in 5 mL lactic acid solution.



For the first week, solutions were refreshed every 24 hours by retrieving the disc and placing it in a new test tube with fresh solution. Solutions without resin discs were not refreshed and served as controls. The discs were gently rinsed with deionized water before re-immersion. After removal of the disc, solutions were prepared for ion readings with the addition of 2% (0.1 mL) ionic strength adjustment buffer (ISA)/Ca sample preparation solution (KCl) to optimize measuring conditions as instructed by the manufacturer. Readings were taken using a calcium-selective electrode (model Ca 800, WTW, Weilheim, Germany). Direct measurements were of membrane potential in millivolts. Prior to a reading, the test tube was agitated to disperse ions evenly. The electrode was immersed in the solution and allowed to equilibrate for 60 seconds before a reading was taken. After recording, the electrode was rinsed with deionized water and dried before testing the next specimen. Between measurement sessions, the electrode was stored in a diluted standard solution in accordance with manufacturer directions. An example of the specimen array and electrode measurement setup is shown in Figure 2. Test tubes remained



capped at all times except when transferring discs or making measurements to avoid evaporation and environmental influences.

**Figure 2.** Specimen array and electrode measurement setup



After the first week of measurements, the discs were again transferred to new tubes with fresh solution. They were allowed to soak an additional five weeks with measurements taken every week as described above. Sample preparation solution was only added before the first set of measurements. Between the weekly measurement sessions, the electrode was rinsed with deionized water, dried, and stored dry with a protective cap in place according to manufacturer instructions.

In order to convert membrane potential measurements into concentration, a calibration curve was obtained by measuring membrane potential of a known calcium solution concentration

in the range of interest to the study (0.1 to 10 mg/L). The calcium solution was manufactured by the same company, WTW.

### **Shear Bond Strength**

Thirty nine extracted human premolars were used for the bond strength portion of the study. IRB approval for the use of the teeth was obtained from the Office of Research Compliance, Marquette University, Milwaukee, Wisconsin (Protocol #HR-1913). Extracted teeth were collected by staff in the Surgical Sciences department of Marquette University School of Dentistry. The teeth were cleaned of debris under running water with a toothbrush, then stored in distilled water until testing. The teeth were randomly assigned to one of three groups; TransBond LV control (no NovaMin added), TransBond LV with 15% NovaMin, and TransBond LV with 22.5% NovaMin. Groups were chosen based upon analysis of calcium ion release results.

Prior to bonding, roots were removed below the cemento-enamel junction using a high speed dental handpiece and diamond bur with water spray. The bonding protocol was followed according to manufacturer instructions, outlined below:

1. Prophyl teeth with an oil-free pumice.
2. Rinse thoroughly with water.
3. Air dry with oil- and moisture-free air source.
4. Apply etching gel for 15 seconds.
5. Rinse thoroughly with water for 15 seconds.
6. Air dry with oil- and moisture-free air source.
7. Apply a thin, uniform coat of primer to the tooth surface.
8. Apply a small bead of resin onto the bracket base and spread over the base using the dispensing tip.
9. Place the bracket onto the tooth surface, position, press, and remove excess flash.

10. Light cure from the mesial and distal for 10 seconds each.

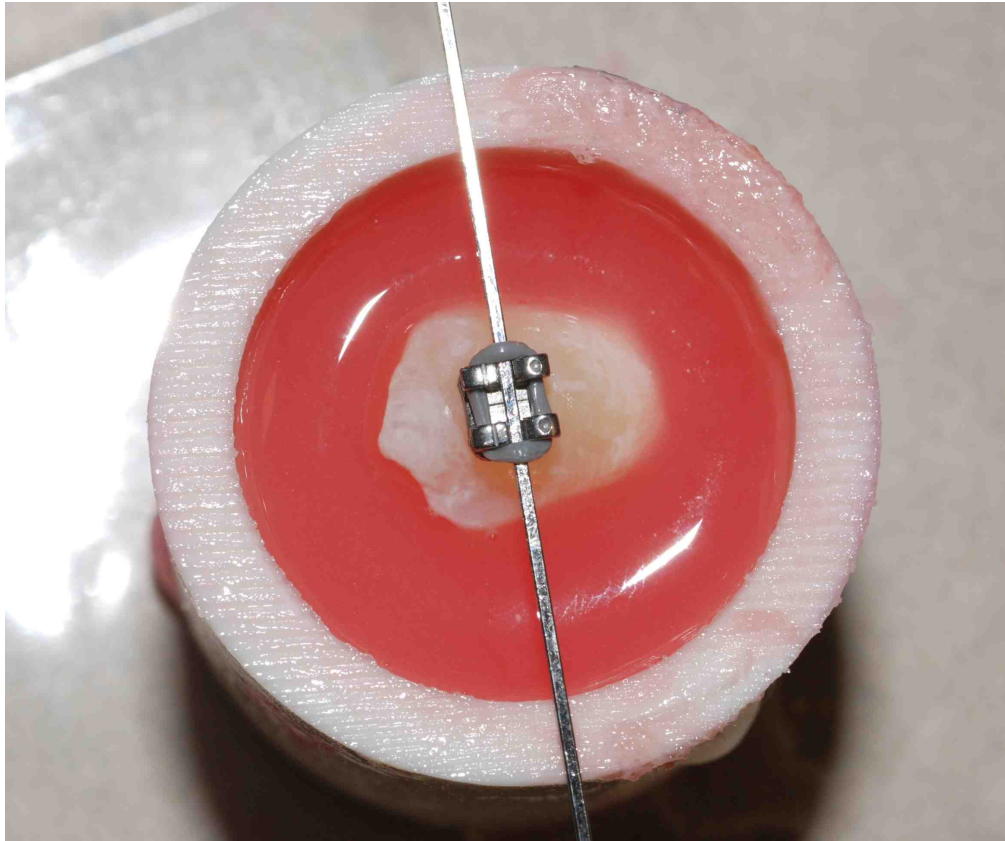
All materials used were from 3M Unitek, including acid etch gel (Unitek 35% phosphoric acid), primer (TransBond XT), brackets (Victory Series upper first premolar, 0° tip and 0° torque) and curing light (Ortholux LED). The bonding materials and instruments used are shown in Figure 3. The curing light remained on the charging base between uses and was tested after bonding each group using a radiometer to ensure consistent light levels.

**Figure 3.** Bonding materials and instruments setup



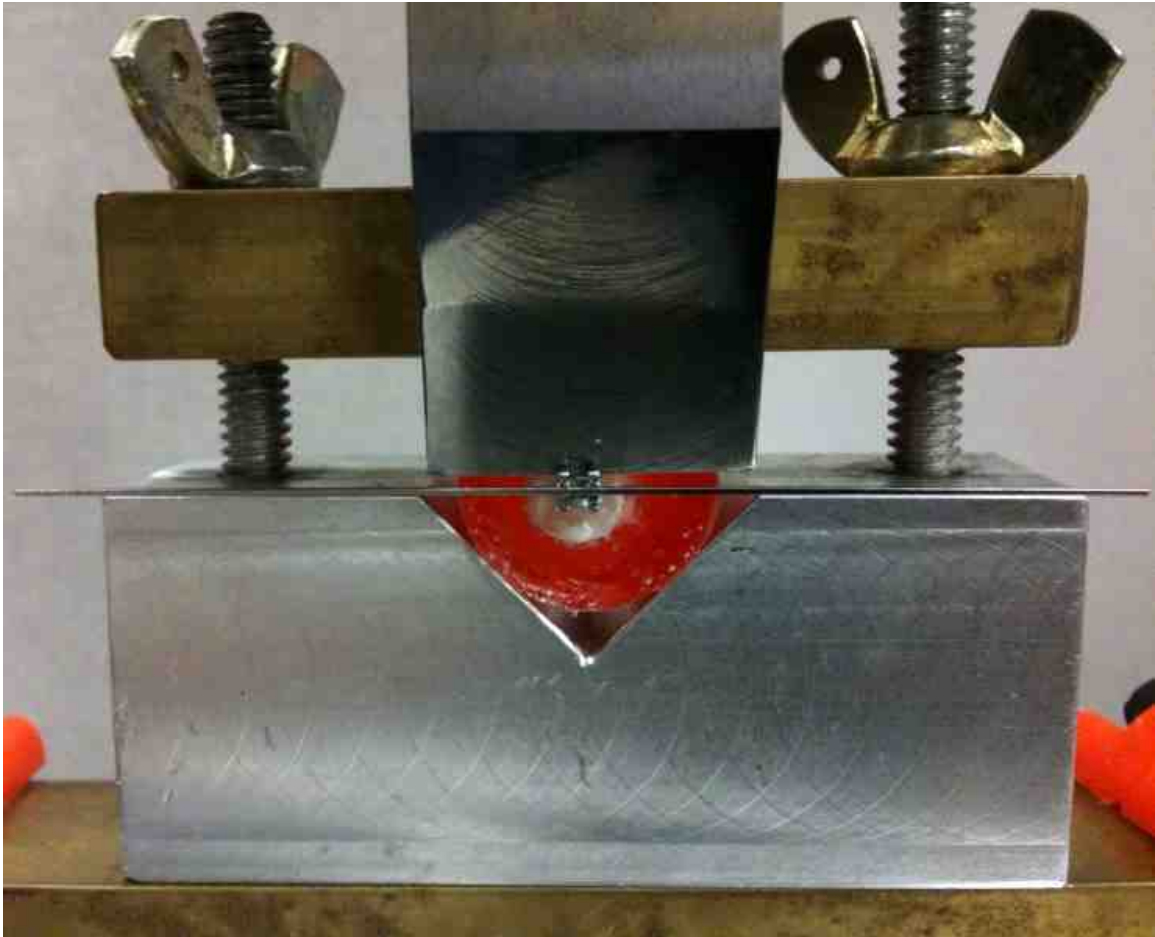
After bonding, the teeth were mounted in acrylic cylinders using a PVC mold. A 0.021” by 0.025” stainless steel wire was ligated to the bracket and extended beyond the top of the mold to aid in correct placement and hold the teeth while the acrylic set fully, as shown in Figure 4. After setting, the support wire was removed and the cylinders with teeth embedded were retrieved from the molds. Specimens were stored in distilled water for 24 hours at 37°C prior to bond strength testing.

**Figure 4.** Acrylic mounting setup



For testing, specimens were mounted in an Instron (Norwood, MA) universal testing machine as shown in Figure 5. A 0.021" by 0.025" stainless steel wire was placed in the bracket slot during mounting to help ensure even contact with the debonding wedge. The wedge was placed as close as possible to the bracket base to maximize the shear component of the debonding force. Brackets were debonded at a crosshead speed of 0.5 mm/min. After debonding, maximum force was recorded in kgf, which was then converted to MPa by multiplying by  $9.8 \text{ m/s}^2$  and dividing by the bracket base area ( $10 \text{ mm}^2$ ).

**Figure 5.** Instron testing machine with specimen in place



Adhesive Remnant Index (ARI) score was assessed for each tooth under a Spencer optical stereomicroscope (American Optical Corp., Buffalo, NY) with external illumination. Values given were as outlined in the literature (Artun and Bergland 1984) as follows:

- 0, no adhesive left on the tooth.
- 1, less than half of the adhesive left on the tooth.
- 2, more than half of the adhesive left on the tooth.
- 3, entire adhesive amount left on the tooth with an impression of the bracket mesh.

Statistical analysis of the bond strength data was processed using one-way analysis of variance (ANOVA) to determine statistically significant differences between the groups. In

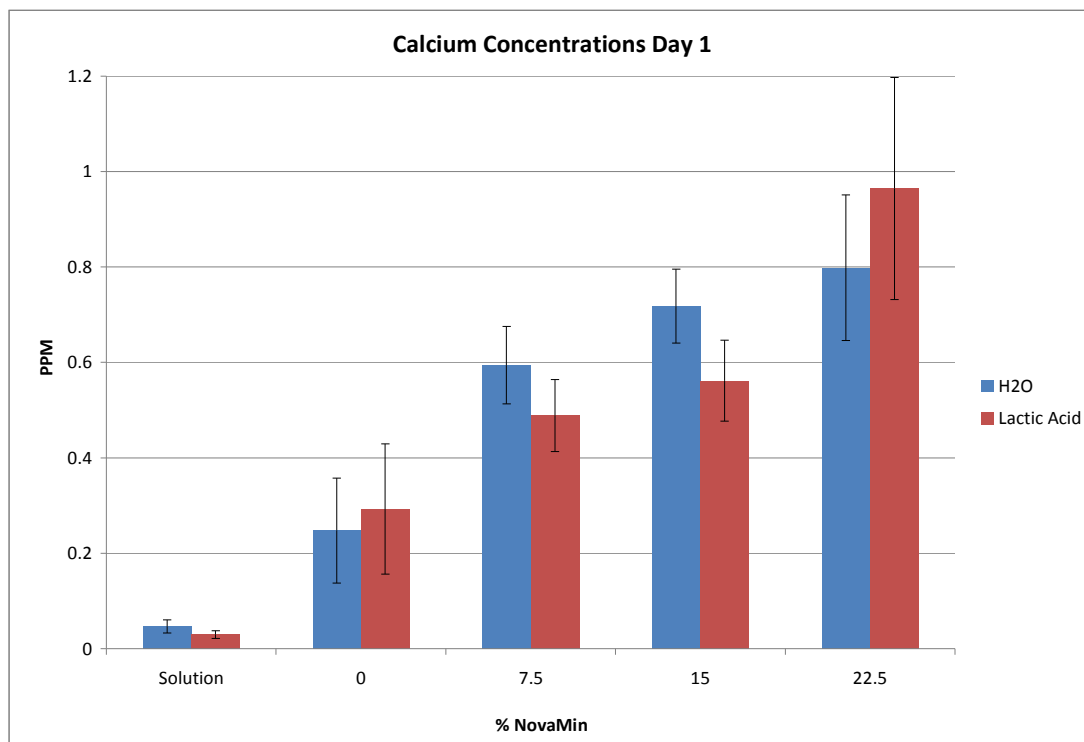
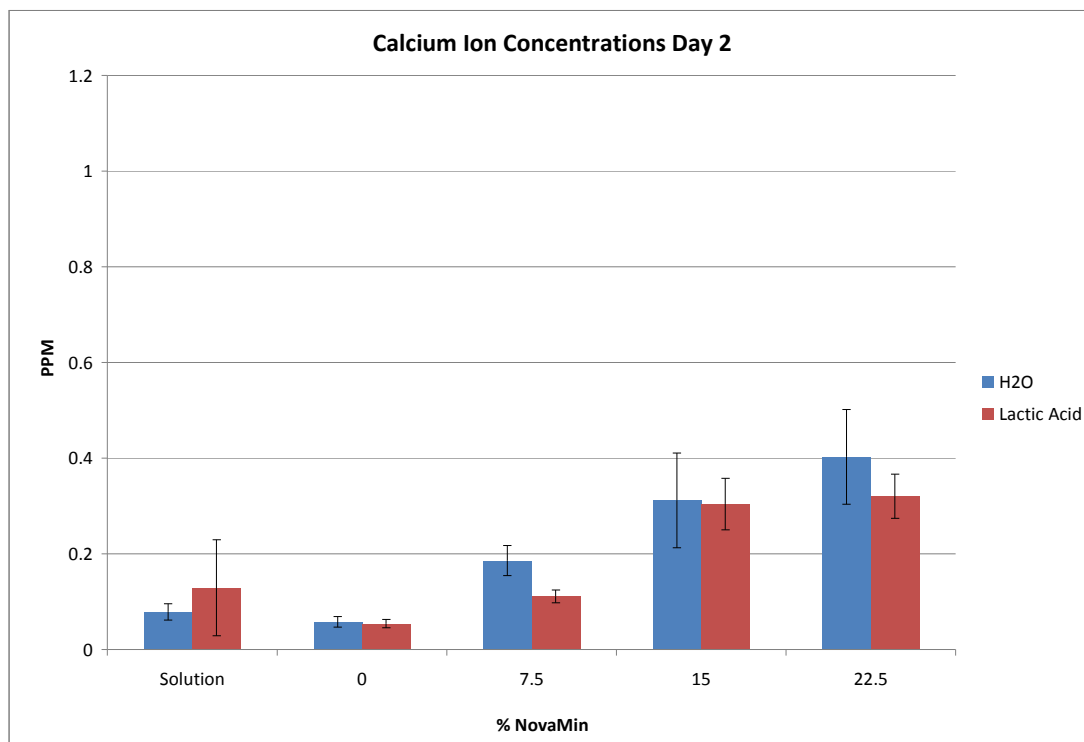
addition, a Weibull analysis was used to determine bond strength reliability. For ARI scores, a Kruskal-Wallis test was used to determine differences between the groups.

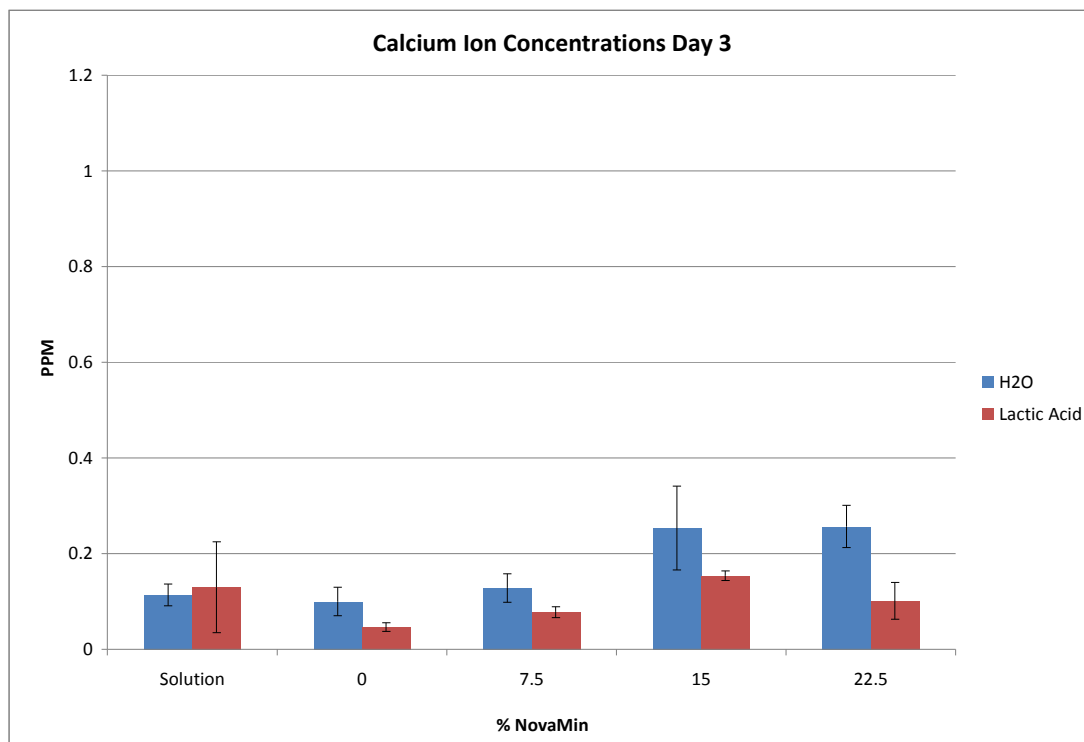
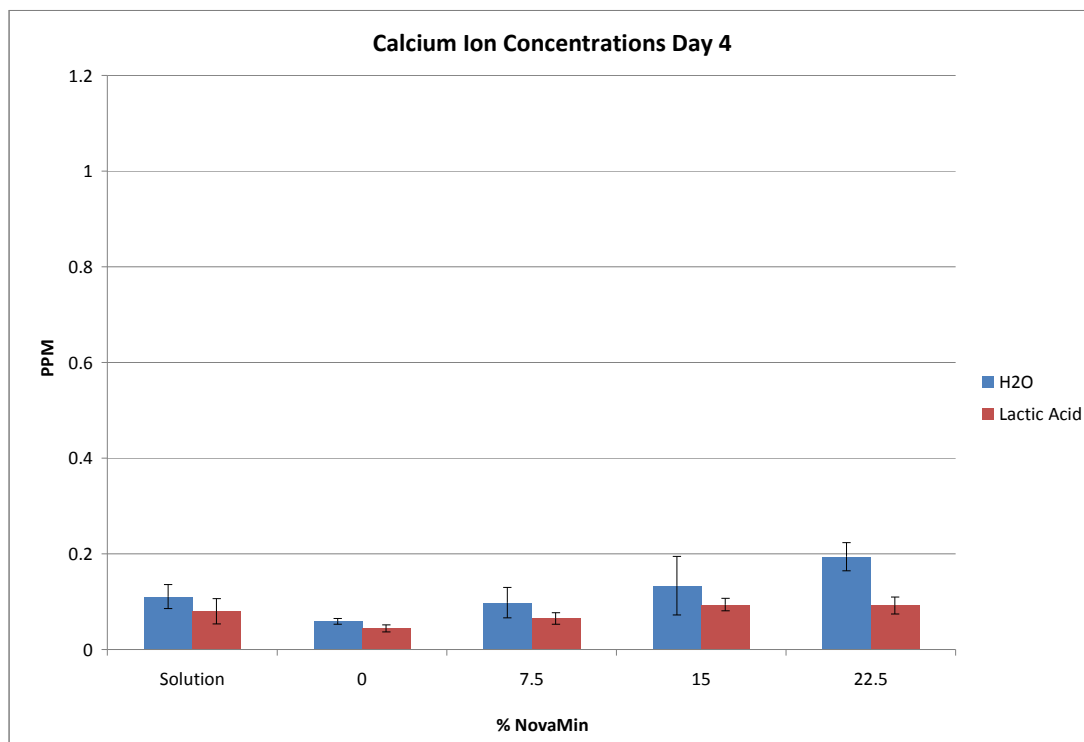
CHAPTER IV  
RESULTS

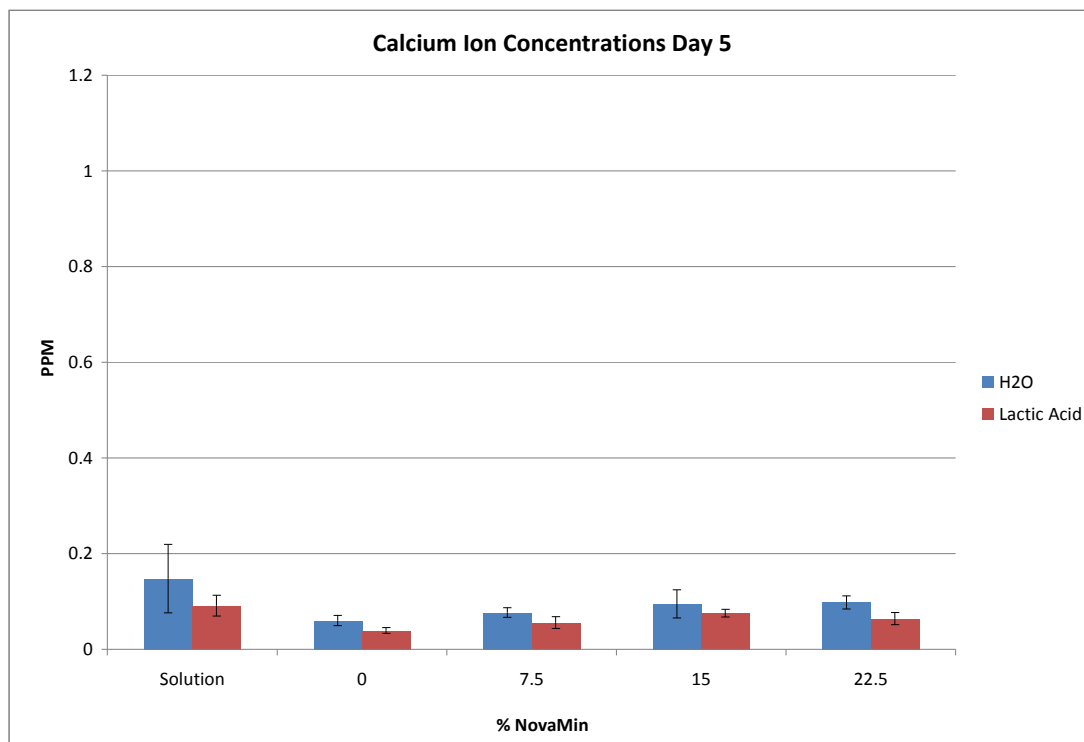
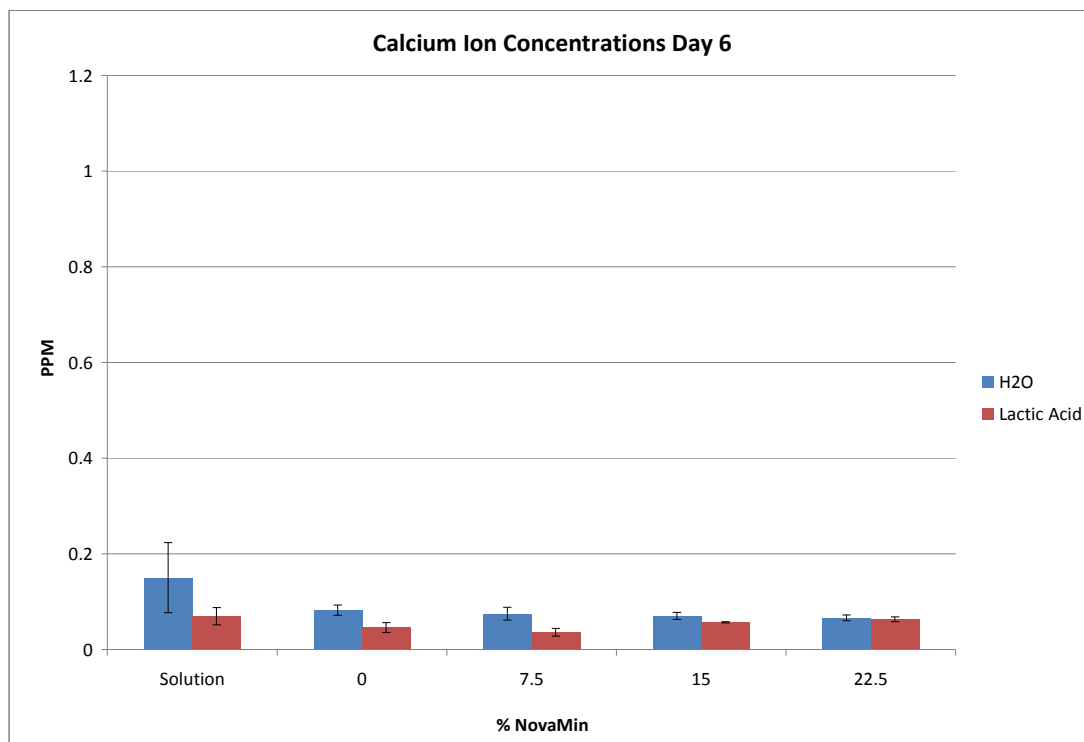
### **Calcium Ion Release**

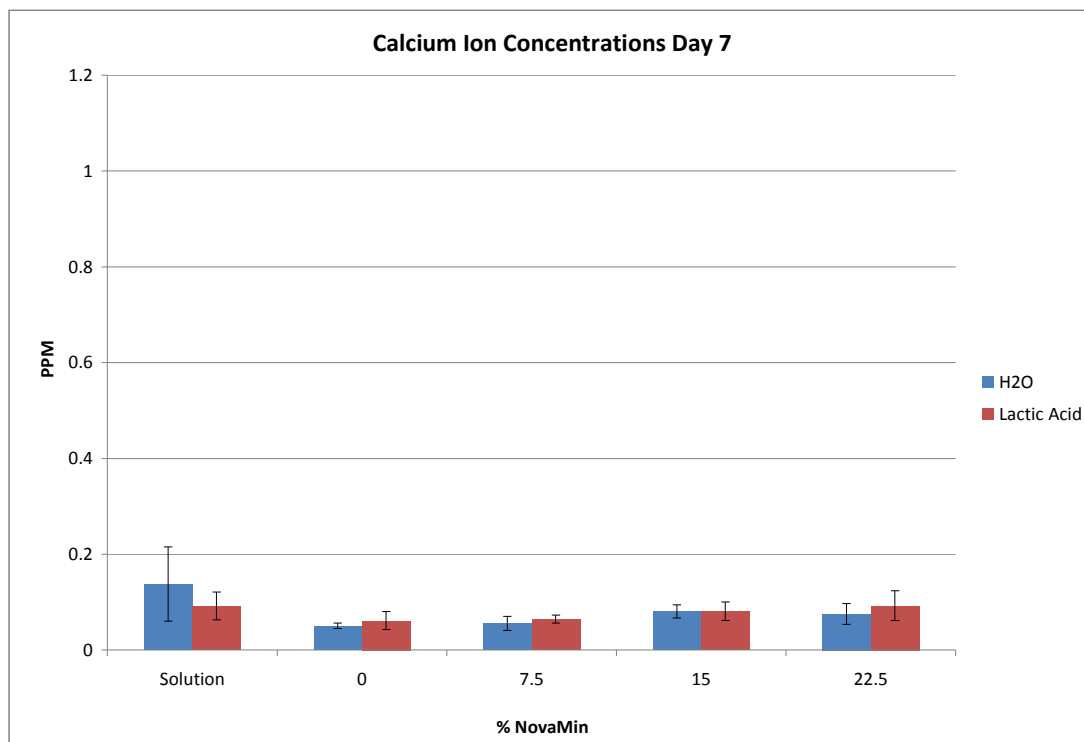
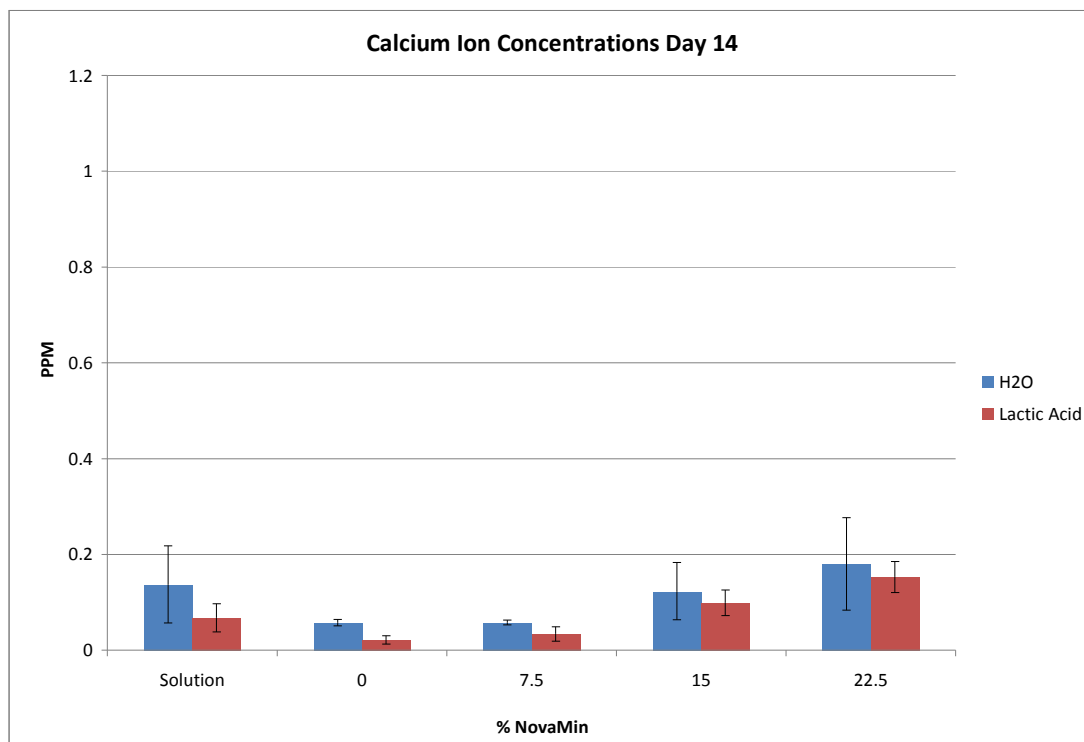
Results for calcium ion release are shown in Figures 6 through 21. Figures 6 through 17 show mean concentration values and standard deviations for each day measured. The values are grouped according to weight percentage of incorporated NovaMin powder for comparison of the differences between the two solutions used. Figures 18 and 19 show concentration changes over time for the first week when solutions were being changed every 24 hours. Figures 20 and 21 show the changes over time for weeks 2 to 6 while the solutions were unchanged.

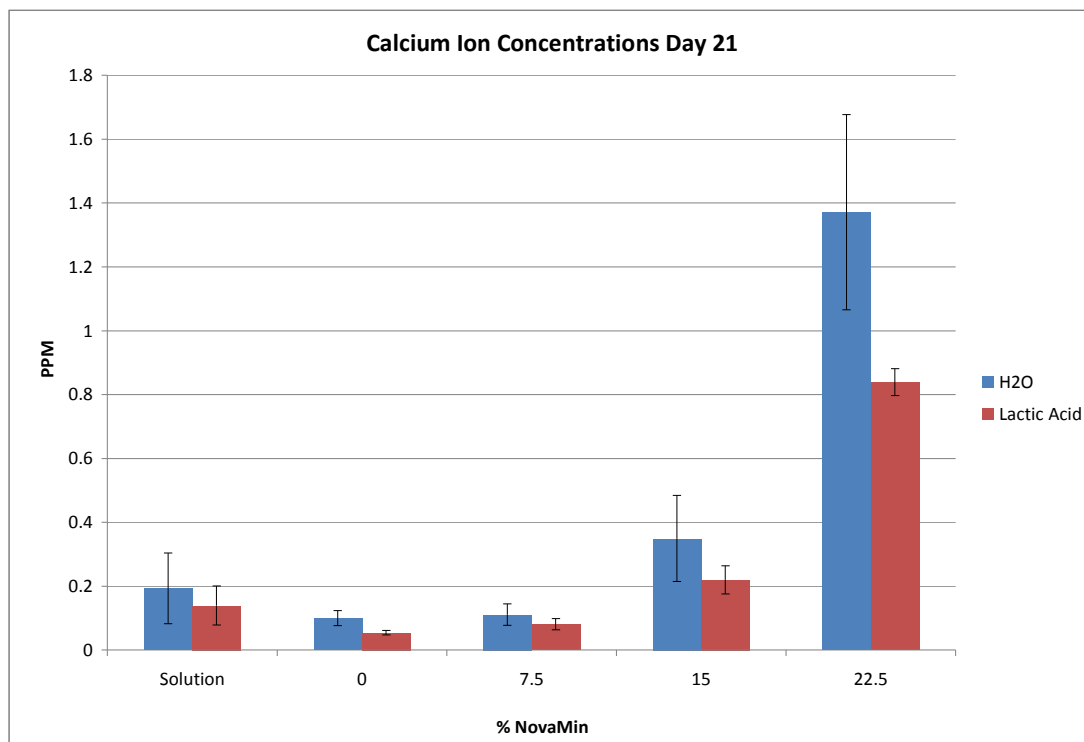
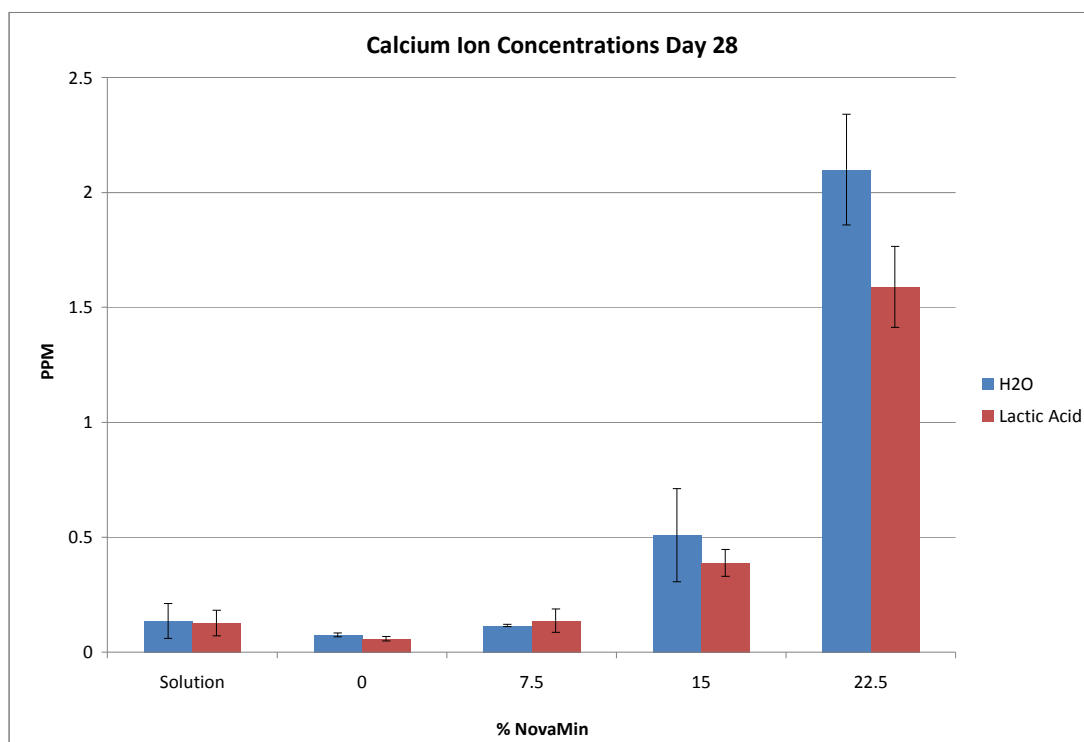


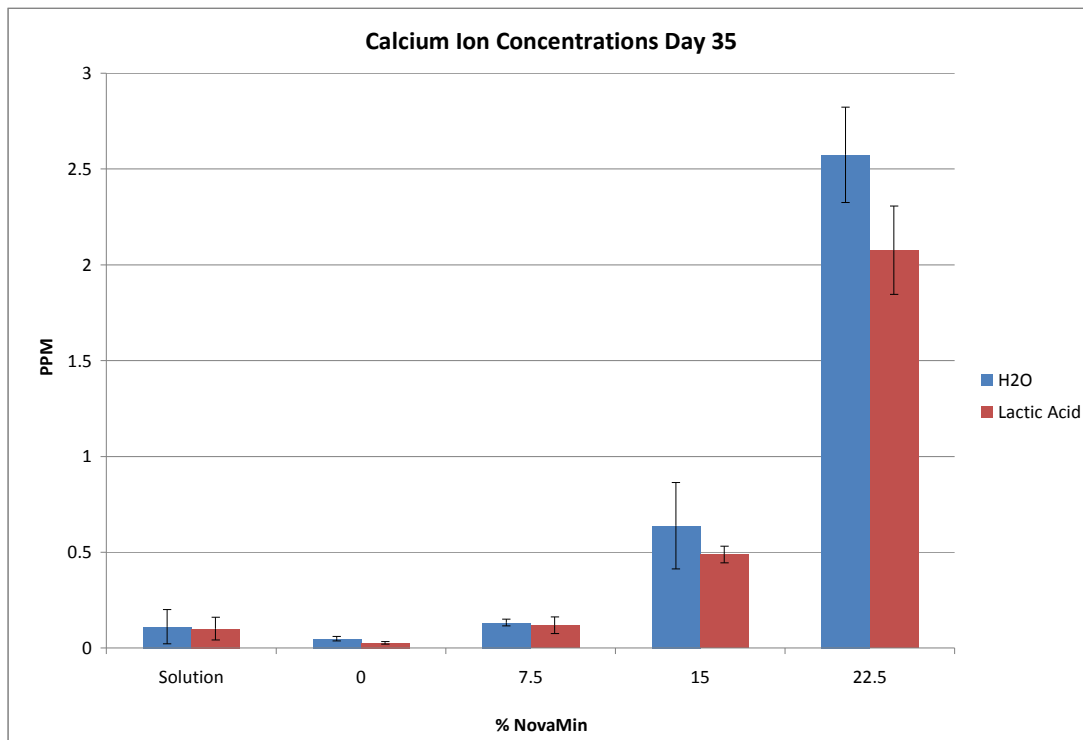
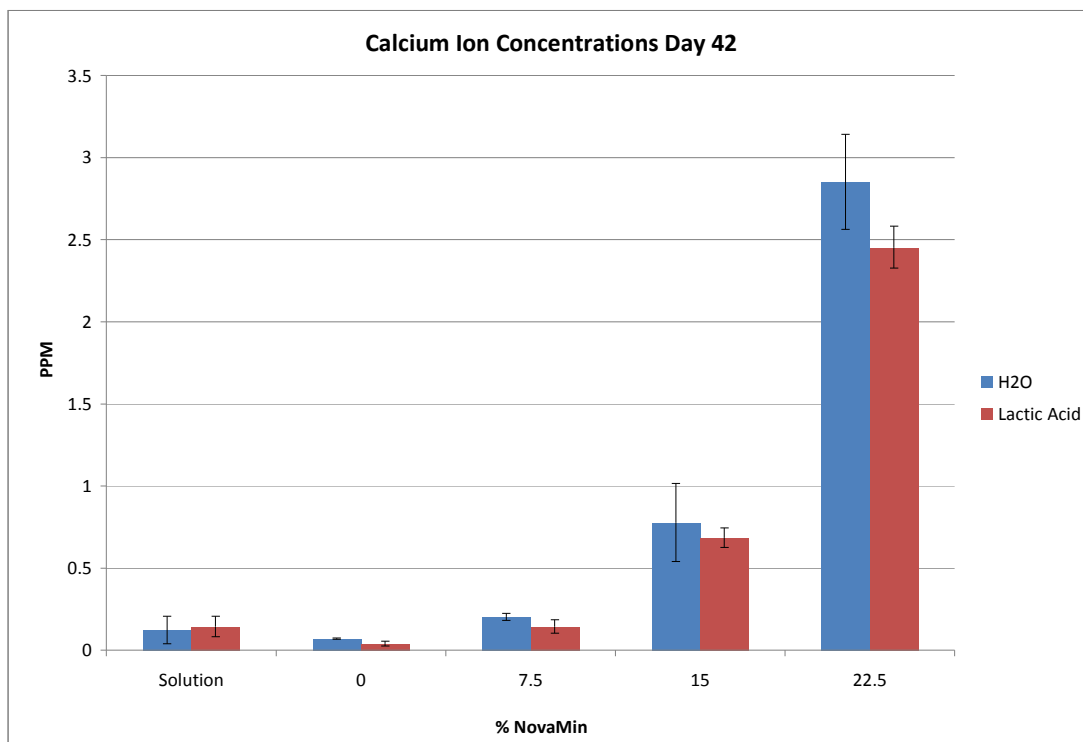
**Figure 6.** Calcium concentrations day 1**Figure 7.** Calcium concentrations day 2

**Figure 8.** Calcium concentrations day 3**Figure 9.** Calcium concentrations day 4

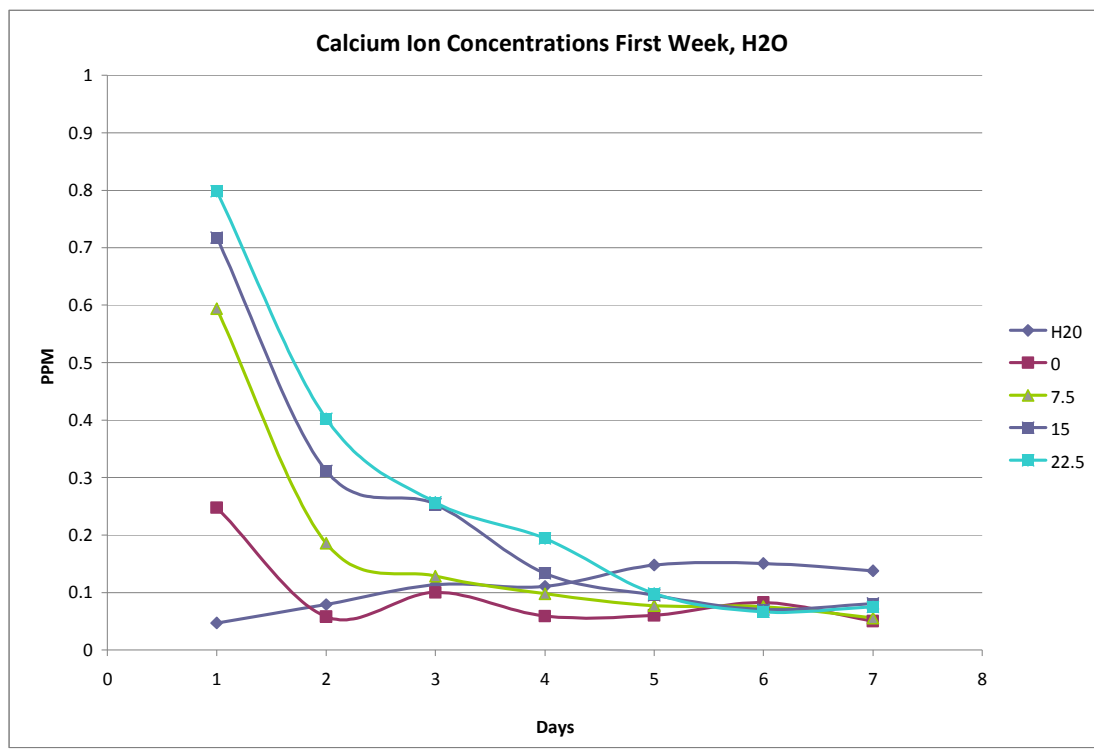
**Figure 10.** Calcium concentrations day 5**Figure 11.** Calcium concentrations day 6

**Figure 12.** Calcium concentrations day 7**Figure 13.** Calcium concentrations day 14

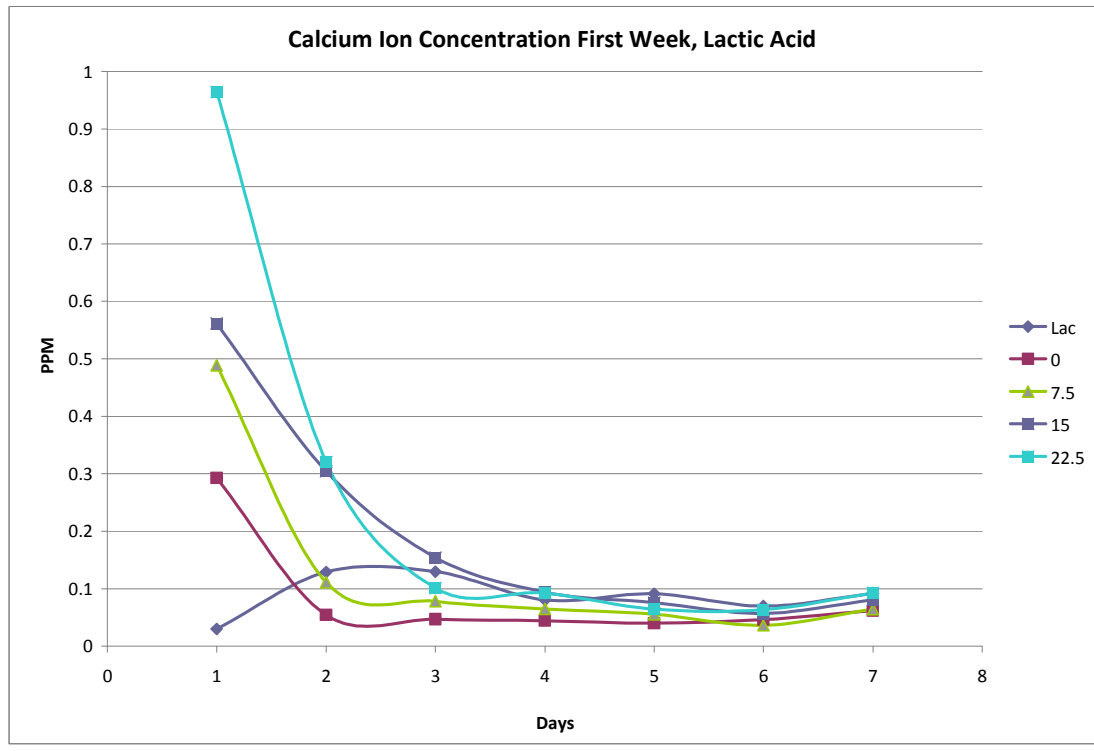
**Figure 14.** Calcium concentrations day 21**Figure 15.** Calcium concentrations day 28

**Figure 16.** Calcium concentrations day 35**Figure 17.** Calcium concentrations day 42

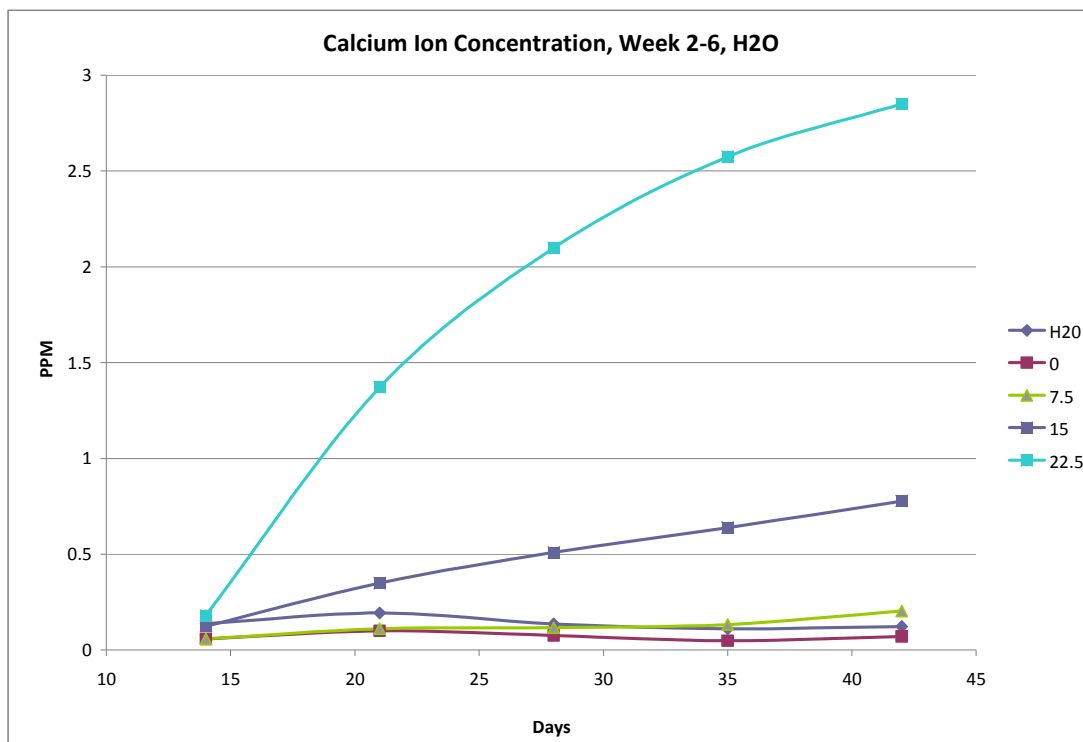
**Figure 18.** Calcium concentrations in deionized water, first week



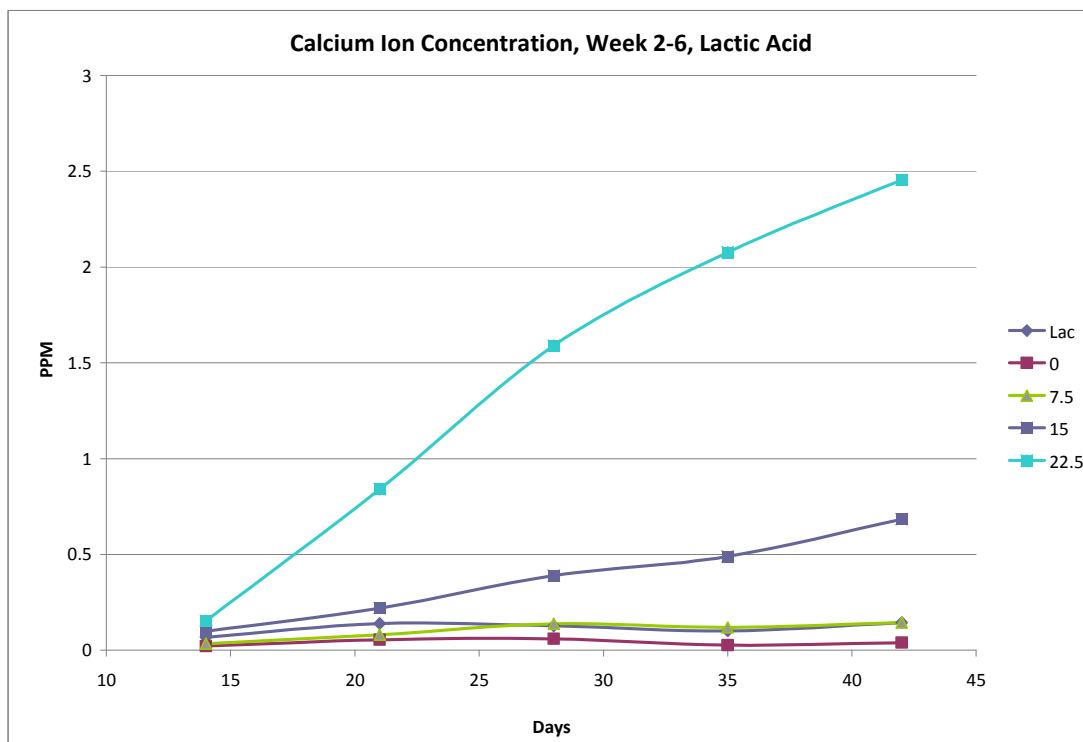
**Figure 19.** Calcium concentrations in lactic acid, first week



**Figure 20.** Calcium concentrations in deionized water, weeks 2-6



**Figure 21.** Calcium concentrations in lactic acid, weeks 2-6





### Shear Bond Strength

Mean shear bond strength values are shown in Table 1. Standard deviation, minimum values and maximum values for each resin tested are also shown.

**Table 1.** Shear Bond Strength Results

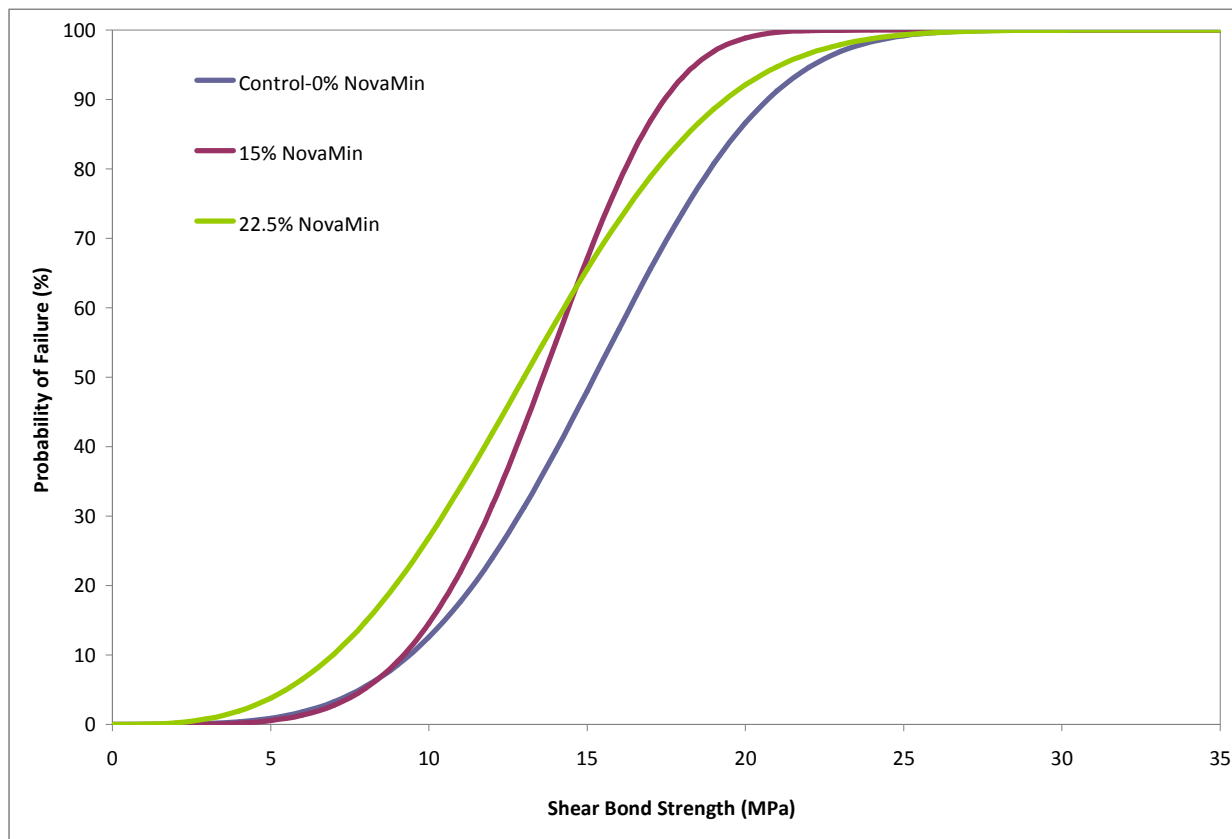
Group	Bond Strength (MPa)				
	Mean	Standard Deviation	Minimum	Maximum	Range
TransBond LV	15.13	4.18	7.99	23.25	15.26
TransBond LV + 15% NovaMin	13.55	2.84	9.26	18.76	9.50
TransBond LV + 22.5% NovaMin	13.27	4.34	8.60	23.11	14.51

One-way ANOVA analysis of the results revealed there was no statistical difference between the three groups (P=0.424).

The Weibull analysis is summarized in Table 2 and Weibull graphical results are shown in Figure 22.

**Table 2.** Shear Bond Strength Weibull Analysis Results

Group	Weibull Modulus ( $\beta$ )	Characteristic Strength ( $\alpha$ ; MPa)	Probability of Failure at 7.8 MPa (%)	Shear Bond Strength (MPa) at 10% Probability of Failure	Shear Bond Strength (MPa) at 90% Probability of Failure
TransBond LV	3.9	16.7	5.0	9.4	20.7
TransBond LV + 15% NovaMin	4.8	14.7	4.6	9.2	17.4
TransBond LV + 22.5% NovaMin	3.0	14.7	13.8	7.0	19.4

**Figure 22.** Weibull Analysis Plot

ARI scores are shown in Table 3. A Kruskal-Wallis test showed significant differences existed between the groups ( $P=0.009$ ). Further analysis with Mann-Whitney tests showed the TransBond LV alone to be statistically different from both NovaMin groups. P-values were 0.003 and 0.028 for comparison of TransBond LV to the 15% and 22.5% groups, respectively.

**Table 3.** Adhesive Remnant Index Scores

Group	ARI score			
	0	1	2	3
TransBond LV	8	4	1	0
TransBond LV + 15% NovaMin	1	6	6	0
TransBond LV + 22.5% NovaMin	3	5	5	0

## CHAPTER V

## DISCUSSION

### **Calcium Ion Release**

Over the first seven days while solutions were changed every 24 hours, calcium ion concentration dropped significantly and eventually reached a level below the reference solutions for both deionized water and lactic acid. A possible explanation may be that NovaMin particles exposed on the surface of the composite resin reacted rapidly and were depleted. Particles dispersed in the matrix would require more time to react and diffuse ions to the surface and into the solution. The observation that levels fell below reference solution values could possibly be explained by noting that the TransBond LV samples without incorporated NovaMin consistently tested below reference throughout the experiment. The resin itself may have components that absorb and sequester calcium ions, but this was not tested and cannot be proven by the results of the study. Another possible explanation may be that the solutions were contaminated slightly by the standard calcium solution the electrode was stored in since they were always the first to be tested on any given day. However, the protocol of thorough rinsing and drying of the electrode with deionized water before each measurement should have prevented any contamination between samples.

From weeks two to six, while the resin discs were allowed to soak without changing the solution, calcium ion concentrations increased significantly for the 15% and 22.5% samples. Over time, the NovaMin particles within the resin matrix may have reacted and diffused ions to the surface and into the solution. This trend has some support in the literature. O'Donnell et al. (2008) tested resin discs with incorporated particles of ACP and found calcium levels in unchanged saline solution increased slowly but consistently over a total of six months.

The general trend during this time period, except for the 22.5% sample on day one, was for ion concentrations to be higher in deionized water than in lactic acid at pH~5. This result is unexpected, since research on NovaMin has shown higher values at lower pH. Cerruti et al. (2005) studied the reaction of pure NovaMin particles in differing solutions and found calcium

ion release was lowest in deionized water. It was explained that due to an increase in pH in the earliest period of dissolution of the particles, calcium release is slower due to a formation of calcium salts which are less soluble at higher pH. During that study the pH of the water solution increased from 5.8 to 10 in the first 30 seconds of the reaction. The increase in pH, as previously described, is due to an exchange of sodium ions in the particles with hydrogen ions in solution (Burwell et al. 2009). No acidic solutions were used in that study, however, and all were buffered to keep pH relatively constant except in the deionized water. The current study used an unbuffered lactic acid solution and may have been subject to pH increases.

The highest calcium ion concentration reached in this study was 2.85 PPM, far below values reported in the literature. Levels in the Cerruti et al. (2005) study were close to 50 PPM in water after three days. In that study, 0.3 g of particles were immersed in 200 mL of solution. At a weight percent of 22.5, each resin disc in this study contained close to 0.1 g particles and was in only 5 mL solution. However, samples were not continuously stirred or agitated as in other studies (Cerruti et al. 2005, O'Donnell et al. 2008, Langhorst et al. 2009). In designing a model for intraoral caries conditions, it can be argued that both a static and dynamic model can be applied, given the dynamic nature of oral fluids and the static nature of plaque colonies.

The only other study found that tested ion concentrations with resin discs was O'Donnell et al. (2008). At a similar time point of two months, calcium ion concentrations reached 0.6 mmol/L with a 40% mass fraction of ACP particles in 100 mL of buffered saline solution. The value in the current study, 2.85 PPM calcium, converts to 0.07 mmol/L. The resin discs used in that study were almost twice the diameter as the current study, however.

Another difference in this study is the use of the calcium selective electrode. It was chosen as a measurement method due to ease of use and compatibility with existing equipment. Similar equipment has been used extensively in the dental field and has proven durable and reliable for continuous, high-volume use (Bowers et al. 1986). Other studies have used

spectrophotometric methods (Cerruti et al. 2005, O'Donnell et al. 2008). Although this difference in measurement techniques should not affect results, it presents an additional variable in making comparisons.

### **Shear Bond Strength**

No significant difference was found in shear bond strength between the three groups tested. This is a significant finding considering comparative literature results. Aegis Ortho, the only commercially available ACP-containing composite, has been shown in several studies to have significantly lower bond strengths than controls (Dunn 2007, Foster et al. 2008, Uysal et al. 2010).

Bond strength values for the TransBond LV were found to correspond well to reported values of the conventional direct bonding composite, TransBond XT. Foster et al. (2008) showed TransBond XT had a mean shear bond strength of 15.2 MPa, remarkably close to the 15.13 MPa of the LV version used in this study.

According to the manufacturer, TransBond LV is a nanofilled resin designed for indirect bonding and lingual retainer bonding. Due to its low viscosity it is not recommended for direct bonding, which proved somewhat difficult in this study with regard to flash cleanup. It is moderately filled, 65% by weight. The particles consist of 75 nm silica and 5-10 nm zirconia (Cinader and James 2009). NovaMin, as reported earlier, has an average particle size of about 2  $\mu\text{m}$ , and contains a large percentage of silica glass. These properties may allow for the NovaMin particles to act as additional filler without compromising bond strength.

The Weibull analysis shows probability of failure at specific loads and can help determine bond reliability. The 15% NovaMin composite showed the greatest modulus, showing higher reliability than the other two groups. That reliability is also reflected in a lower standard deviation. In contrast, the 22.5% NovaMin composite showed a trend towards more early

debonds with a probability of failure of 13.8% at a bond strength of 7.8 MPa, a level reported in the literature to be clinically acceptable (Artun and Bergland 1984). This is likely because of a higher standard deviation than the 15% group.

ARI scores were found to be statistically different for the TransBond LV group compared to the two NovaMin groups. Overall, there was less adhesive remaining on the teeth when the resin was used alone. No published results for TransBond LV could be found for comparison of this result. Although not statistically significant, slightly lower mean bond strengths were observed when NovaMin particles were incorporated into the resin matrix. These particles may have a lower binding affinity to the matrix due to their composition and may allow for a loss in cohesiveness of the composite, resulting in a different mode of failure.

CHAPTER VI  
SUMMARY AND CONCLUSIONS



The results of this study have shown that NovaMin, when incorporated into a commercially available low-viscosity resin composite in sufficient amounts, can release low levels of calcium ions over time in both deionized water and lactic acid. Additionally, no statistically significant difference was found in shear bond strength with this modification.

Given these results, and considering the limitations of this study, further investigation could be justified. A custom-designed resin composite with greater ion-releasing capabilities and a higher percentage of NovaMin acting as filler could obtain higher ion release profiles, possibly without adversely affecting bracket bond strength. It could also be used as a filler for a sealant in the same manner.

Additional benefits to such a resin or sealant that could be studied include antibacterial and antiplaque properties, as they have been demonstrated in the literature for NovaMin-containing products currently available (Tai et al. 2006). Local effects on pH of plaque colonies could also be studied since the reaction of NovaMin increases pH (Burwell et al. 2009).

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