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Enhancing Dairy Processing Equipment with Antimicrobial Silicone Resin: The Effects of Zinc Oxide on Mechanical Properties

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ABSTRACT

Ensuring the quality and safety of dairy products starts with the surfaces that encounter milk, necessitating materials that are smooth, impermeable, and free from any defects that could harbor bacteria. Traditional materials like metals have been found lacking due to issues like corrosion and bacterial contamination. In response, the dairy industry has turned to silicone resin for its corrosion resistance and ability to maintain the freshness of milk. A challenge with silicone and similar polymers is their propensity to develop biofilms from continuous exposure to bacteria, which can lead to infections spreading during the milking process. Prior work by authors has established the convence of ZnO antimicrobial properties when mixed into silicon resins. The goal of this paper is to assess how ZnO affects the resin's elasticity and hardness, which are critical for the reliability and cleanliness of dairy equipment. Findings show that while lower concentrations of ZnO did not affect the physical properties significantly, a 4% concentration increased its stiffness. This suggests a promising path for producing dairy processing materials that are not only mechanically robust but also inherently antimicrobial, potentially revolutionizing hygiene standards in dairy farming without compromising equipment performance.

Research Highlights

- **Enhancement of Dairy Processing Equipment:** The study highlights the crucial role of maintaining the integrity of milk contact surfaces to ensure product quality and safety in dairy processing. By incorporating zinc oxide (ZnO) into silicone resin, traditionally used in milking liners, the research aimed to enhance the material's antimicrobial properties without compromising its essential mechanical characteristics. This innovation addresses the limitations of traditional materials like metals, which can suffer from corrosion, off-flavors, and bacterial contamination.
- **Innovative Integration of ZnO for Antimicrobial Properties:** The research focused on integrating ZnO, known for its antimicrobial capabilities, into silicone resin at varying concentrations (1%, 2%, and 4% w/w). This strategic addition aimed to combat the formation of biofilms on milking liners, a common issue leading to infection transmission in dairy farms. The study meticulously evaluated the impact of ZnO on the elasticity and hardness of the silicone resin, crucial for the durability and functionality of dairy processing equipment.
- **Statistical Significance in Mechanical Properties:** While the addition of ZnO up to 2% concentration did not significantly alter the silicone's hardness or stiffness, a notable finding was that a 4% ZnO concentration resulted in a statistically significant increase in material stiffness. This outcome suggests a tangible improvement in the material's physical properties at higher ZnO concentrations, potentially enhancing the performance of dairy processing applications.
- **Optimization of Material Properties through Advanced Fabrication Techniques:** The study employed a pioneering technique to ensure uniform distribution of ZnO particles within the silicone matrix, addressing a critical challenge in composite material science. The successful integration of antimicrobial properties into silicone resin, validated by the consistent coloration of the fabricated plaques, represents a significant advancement in engineering materials for the dairy

industry, offering a novel solution for improving the hygiene and mechanical integrity of milking equipment.

Keywords

Food Technology, Engineered Materials, Dairy Processing Equipment, Zinc Oxide, Milking Liners.

Introduction

The dairy industry is a critical sector that requires meticulous attention to the quality and safety of milk and milk products. This necessitates maintaining the integrity of milk and milk product contact surfaces, which should be smooth, impermeable, free from cracks and fissures, non-porous, corrosion-resistant, durable, non-contaminant, and easily cleanable to meet sanitary design criteria [1]. Traditional materials like metals and their alloys, including aluminum, copper, and iron, have been widely used in dairy processing equipment but have shown limitations due to the development of off-flavors, material discoloration, corrosion, and the potential generation of hazardous bacteria [1,2].

To overcome these challenges, silicone resin has emerged as a preferred material for milking liners in the dairy industry, attributed to its superior corrosion resistance, which ensures the taste of fresh milk is not compromised and guarantees equipment reliability and product purity [1]. However, despite its advantages, silicone resin is prone to biofilm development due to continued contact with bacteria from the animals, which poses a risk of spreading bacteria as the liners are used for multiple milkings [1,3].

Biofilms are complex structures formed by microorganisms that adhere to surfaces and are encased within a protective matrix. These biofilms are significant because they can harbor bacteria that lead to the spread of diseases among cows during the milking process [4]. A particular concern in this context is mastitis, an inflammatory condition of the mammary gland primarily caused by bacterial infections. Mastitis can result from the transfer of bacteria between cows via contaminated milking liners, leading to significant economic losses for dairy farmers due to decreased milk production, altered milk quality, and increased treatment and management costs [3,5].

Engineered materials, often composite in nature, are developed through the integration of multiple material phases to achieve mechanical properties superior to those of each individual constituent [6]. These composites typically combine a strong matrix phase with a tougher, yet less flexible, fiber phase to enhance structural rigidity and strength. Silicone elastomers, a key example of such materials, gain their mechanical integrity from both chemical crosslinking and filler reinforcement, necessitating effective interaction between the polymer molecules and filler particles for optimized reinforcing effects [7,8].

In the context of dairy farming, silicone liners undergo significant repetitive stress due to the combination of vacuum and compression during the milking process. These mechanical stresses, influenced by the vacuum, liner properties, liner on-time, and teat anatomy, lead to changes in teat length and tissue thickness as a result of the forces exerted by the vacuum and the collapsing liner [9-11]. The interaction between these factors can result in high pressure at the most incompressible part of the teat end, where the liner encounters the greatest resistance [12].

Despite the critical role of liners in the milking process, there is a lack of consensus on what differentiates an adequate liner from an inferior one. Liner design remains largely empirical, highlighting the need for consistent mechanical properties in the material along with a method for measuring the load distribution on the teat. Such a method could facilitate the quantification of liner characteristics and the development of customized liners for specific breeds or herds. It could also be used to evaluate existing liner designs, explore the impact of changing liner properties, and investigate the effects of liner aging. Understanding teat pressures is essential for establishing boundary conditions in analytical models aimed at predicting tissue stresses [13].

Recent studies have also explored the gelation behaviors and mechanical properties of silicone resins for specialized applications, such as constructing breast cancer training models, indicating the versatility and importance of understanding the mechanical behaviors of silicone-based materials [8]. Additionally, research into silicone/epoxy hybrid resins has shown potential for tunable mechanical and interfacial properties, which could be beneficial for the additive manufacture of soft robots and other innovative applications [14]. The thermal stability, mechanical, and optical properties of novel addition-cured composites of silicone resin further demonstrate the ongoing advancements in silicone resin technology, offering enhanced properties for a range of applications [15].

Given these challenges, this experimental project aims to explore the modification of silicone resin used in dairy liners by incorporating zinc oxide (ZnO) as an antimicrobial additive at concentrations of 1%, 2%, and 4% by weight. The objective is to evaluate the impact of ZnO on the mechanical properties of silicone resin, including tensile strength and hardness, and to understand how these modifications can enhance the material's performance in dairy processing applications [1].

Elasticity, the ability of a material to revert to its original shape and size after deformation, is a fundamental characteristic of all materials. This property allows for the formulation of silicones with a broad spectrum of hardness levels, from soft and elastic to hard like ebonite, measured using the International Rubber Hardness or Shore gauges, which range from 0-100 degrees. Studies on various liner designs have indicated that the design of the liner plays a more significant role in influencing milking characteristics than any other factor related to the milking machine, with notable differences observed in liner collapse, incidence of liner-slips, and machine operation times among different liners [5]. The optimal composition for a liner is determined through manufacturer testing, with standard acceptance tests including modulus and tensile testing. Modulus testing measures the stiffness of the silicone resin by determining the tensile stress needed to achieve a specific elongation, such as 300%. This research is significant as it seeks to offer insights into optimizing materials for food contact surfaces in the dairy industry, focusing on enhancing both hygiene and mechanical integrity to improve equipment reliability, product purity, and, ultimately, animal and consumer health.

Materials and Methods

The consistent mechanical properties of milking liners are crucial, as they represent the sole point of direct contact between the machine and the cow. The efficacy of the milking machine is significantly influenced by the interaction between the teat and the liner. With hundreds of commercial liners available globally, the complexity of their chemical composition, including up to twenty different ingredients blended homogeneously, is notable. Furthermore, to ensure optimal milking performance and reliability, it is essential for liners to maintain high standards in dimensions and physical attributes across different batches. Figure 1 shows the laboratory-scale technique that allowed for (a) the even distribution of ZnO particles within the polymer matrix, (b) the shaping of the ZnO-enhanced resin into predetermined molded forms, and (c) the production of cured samples devoid of major air inclusions. Fabricating these composite coupons used a pioneering technique designed to achieve uniform distribution of zinc oxide particles within the composite matrix. This approach addresses a critical aspect of composite material science: the precise positioning and concentration of additives to optimize material properties.

A platinum-cure silicone polymer (Smooth-Sil 950, Smooth-On, Inc., Macungie, PA, USA) was selected as the test material because it is commercially available, has simple processing requirements, and approximates the physical characteristics of the proprietary polymers used in many common dairy liners. Zinc Oxide (ZnO) (ZnO 104, ZoChem, Bradbury, TO, CA) was supplied by Buffalo Technology Group, Ltd (Dimmitt, Texas, USA) as the antimicrobial test agent.

A custom, 5-piece aluminum mold was designed using Fusion 360 (Autodesk, Inc, San Francisco, CA, USA). The mold was designed to simultaneously cast 2, 10 cm by 30.5 cm test plaques measuring 3 mm thick. The 3 mm thickness was selected because it is the measured wall thickness of critical features within the popular Pro-Square dairy liner (Pro-Square DPX CR, IBA Dairy Supplies, Sutton, MA, USA). The 5-piece design and critical features are displayed in Figure 2.

The mold parts were machined from 8 mm by 102 mm rectangle bars of 6061-T6511 extruded aluminum (Online Metals, Seattle, WA, USA) using a Tormach 1100MX CNC Mill (Tormach, Inc, Madison, WI, USA). The most critical features of the mold were the flatness of the interior faces and the thickness of the spacers. To make these features as accurate and planar as possible, a fly cutter (Tormach TTS Super Fly Cutter) with a polished carbide insert (SEHT43AFNN-X83) was used to machine the surface of the parts, and special care was taken when programming tool paths and designing work-holding fixtures. Once machined, the interior faces of the mold were polished to a semi-mirror finish using an aluminum polishing compound and cotton buffing wheels. To prepare for molding, the mold was cleaned with glass cleaner, denatured alcohol, and then coated with mold release (Ease Release 200, Mann Release Technologies, Macungie, PA, USA) and then assembled using 18, 0.25 in x 20 – 0.75 in socket head screws.

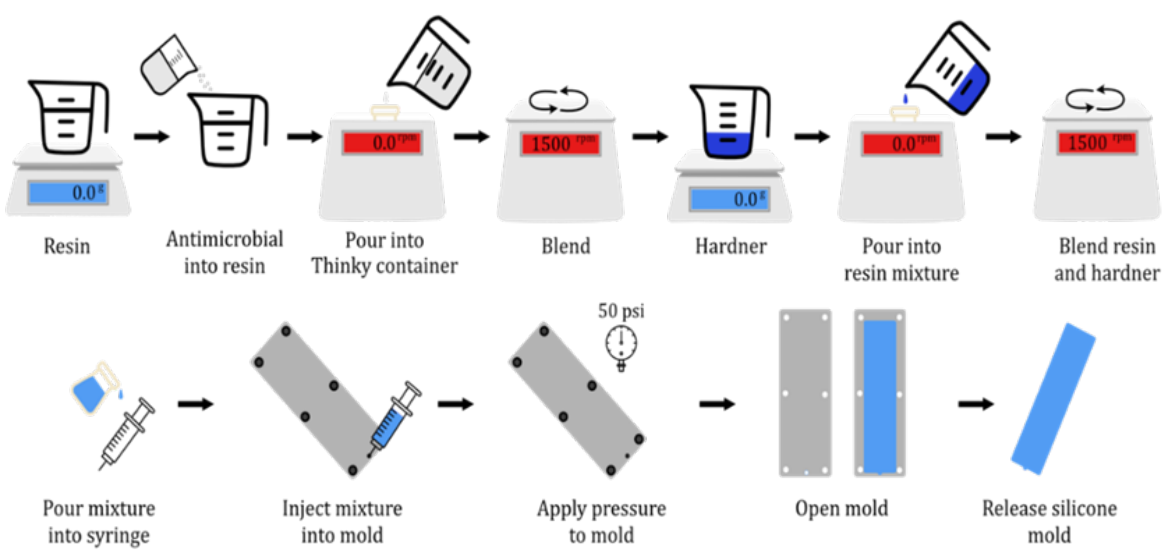
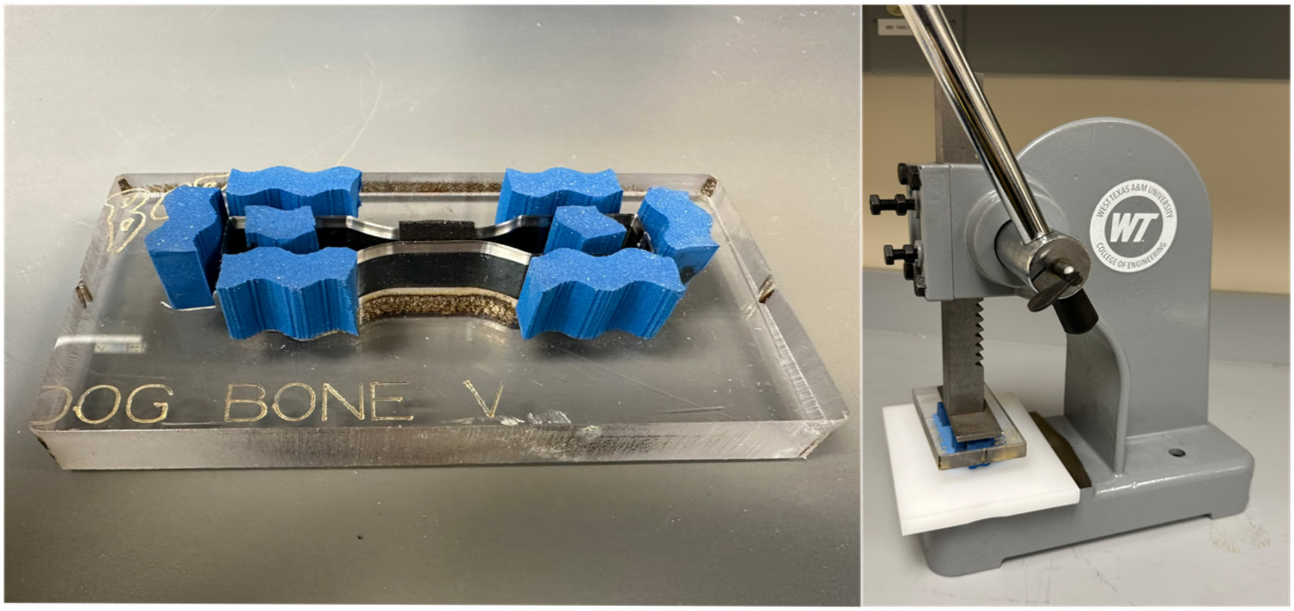


Figure 1: Novel technique for uniform distribution of ZnO particles in polymer matrix.



A B

Figure 2: Steel Rule cutting die (a) and 2-ton arbor press (b) used to die-cut samples for uniaxial tensile test.

The two-part silicone polymer was mixed at a 10:1 resin-to-hardener ratio per manufacturer instructions. The antimicrobial additive, ZnO, was added to the mixture at 0, 1, 2, and 4 percentage by weight basis (*w/w*), where the weight of the additive is expressed as a percentage of the total weight of the polymer. The polymer was mixed in a two-step process to decrease agglomeration without decreasing pot life. The resin (Part A) was first metered into a 300mL HDPE mixing jar then ZnO was added to the jar. This mixture was blended via planetary mixing (ARE 310, Thinky USA, Laguna Hills, CA, USA) for 2 minutes at 1500 rpm. Then, the hardener was added, and the mixture was blended for 2 minutes at 1500 rpm. While blending, the mold was arranged in a bench vise at a 45-degree angle such that the injection port was below the exhaust ports of the mold. Once blended, the mixture was quickly poured into a 300ml barrel syringe with a catheter tip and injected into each cavity of the aluminum mold. Care was taken to inject the mixture under consistent pressure and to allow adequate time for air to evacuate the mold through the top exhaust ports. Once resin fully filled both cavities, the bottom injection ports were sealed with a 0.3125 in x 18 – 1 in bolt. The bolt was screwed 12 mm into the injection port assembly, injecting more resin into the mold cavity and decreasing the volume of air close to the injection ports.

The mold was then placed in a pressure chamber at ambient temperature under 50psi to decrease the number and size of air pockets within the samples. After 24 hours, the mold was removed from the pressure chamber, and the molded samples were released from both cavities of the mold. The samples then underwent a two-step wash in dish soap and deionized water to remove the mold release from the surface. The molded samples were then cut into 44.5 mm by 70 mm coupons with a rotary cutter.

Shore A Hardness Testing (ASTM D2240)

Shore A hardness is a measure of the hardness of a material, typically used for rubber or soft plastics. The Shore A scale gives a numerical value to the hardness, with higher numbers indicating a harder material. The Shore A hardness was taken using a method that approximates ASTM D2240 using a handheld digital durometer (Model No. 3805B, L.S. Starrett Company, Athol, MA, USA) according to ASTM D2240. Two castings were made for each of the 4 treatments. To achieve the minimum thickness required for testing, plies were constructed by stacking two coupons, cut from the same plaque, together to form a hardness test sample. In total, 40 hardness test samples were constructed (10 from each treatment). On each sample, 5 areas were identified that were at least 6.5 mm from the edge and 6.5 mm from each other. A 605 g steel weight was added to the top of the durometer to apply consistent pressure, large enough to fully bury the probe tip in the polymer, during each reading. The weight was balanced laterally during measurement and no additional pressure was applied in the vertical direction. Five readings were taken from each of the 5 areas on each of the 40 samples. The average value of the 25 readings is reported as the sample hardness per ASTM specification.

Tensile Testing (ASTM D638)

One casting was made for each of the 4 treatments. Dumbbell-shaped samples (n=16) were cut from each casting using a steel rule cutting die (ASTM D638-V) and a 2-ton arbor press (Figure 2). Each of the 64 samples was tested in triplicate for a total number of 192 tests (4 treatments x 16 samples x 3 tests per sample).

A UniVert Mechanical Test System (CellScale Biomaterials Testing, Waterloo, ON, Canada) was used to test the elasticity of each sample (Figure 3). Univert software (ver-12.51) was used for data acquisition with a feature to capture sequential images using a scientific camera. The experimental setup and procedure were augmented from the setup of Kundapur and Unnikrishnan [6] and are shown in Figure 3. The load cell used in the setup was a semiconductor strain gauge-based with an accuracy of 0.2% of the rated full-scale load up to 100 N. The load cell was calibrated using a known mass of 100 g provided by the manufacturer. The distance between the grippers was calibrated using a compression spring of known length, which was also supplied by the manufacturer. This distance calibration procedure was performed after every 5 samples, and the height was also independently validated using digital calipers. The grippers were set to 40 mm, and the sample was loaded such that when the grippers were tightened, the midpoint of the sample was aligned with the midpoint between the grippers.

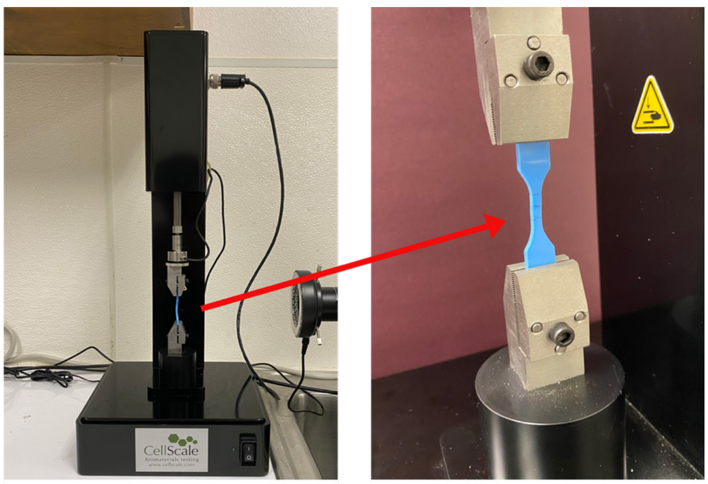


Figure 3: Experimental setup for uniaxial tensile test (a) and inset of setup showing polymer coupon clamped into the fixture (b).

Each sample was tested in 3 successive test replicates with a preload of 0.1 N and time durations set for stretch (30 s), recovery (10 s), and rest (10 s). During testing, strain was limited to a maximum of 50% to ensure that the samples remained within the elastic deformation range. This approach helped avoid any damage to the samples and prevented them from slipping out of the grips due to changes in volume while being clamped. Data was gathered at a frequency of 5 Hz, and images were captured at 1 Hz (Figure 4). The 3 test replicates yielded approximately 765 data points for each sample. The data was analyzed using R-Studio (Posit team, Boston, MA, USA). The strain (denoted as ε) and stress (σ) for each point were calculated using Eqs 1 and 2, respectively. The initial length (L°), displacement (ΔL) and force (F) were captured with the software. The cross-sectional area (A) for each sample was 7.314 mm^2. The strain (independent variable) and stress (dependent variable) for each sample were plotted and a linear regression was generated for each plot (the minimum R^2 value for each of the 64 tests was 0.95 with an average of 0.96). The slope of the regression represents the stiffness of the specimen, which in this linear case is Young's Modulus. The arithmetic mean of the moduli was calculated for each sample per ASTM specification and are shown arranged by treatment in Figure 6. The significance threshold for this study was set at 0.05.

$$\varepsilon = \frac{\Delta L}{L^{\circ}} \quad (1)$$

$$\sigma = \frac{F}{A^{\circ}} \quad (2)$$

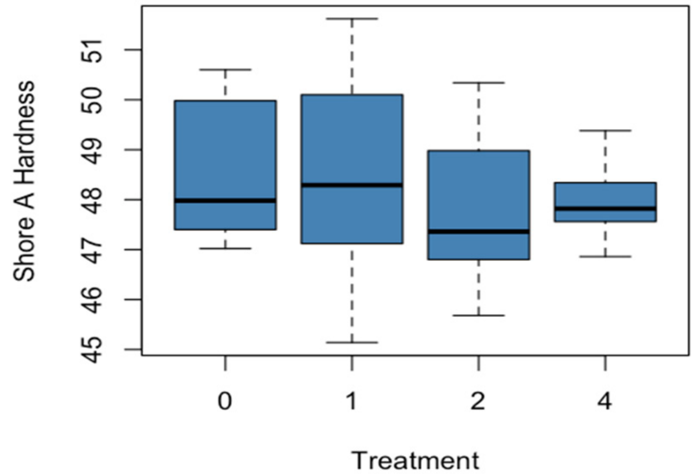


Figure 4: Average Shore A Hardness for treatments of zinc oxide in silicone polymer matrix.

Power Considerations for Tensile Testing

The experimental design for tensile testing was informed by prior studies assessing Young's Modulus in silicone and silicone-composite materials, which typically use 6–15 specimens per group due to the low within-group variance in mechanical testing setups [6,8,14]. In this study, sixteen independently cast specimens were prepared for each treatment group and tested in triplicate, resulting in 48 measurements per treatment and 192 total modulus tests. A post-hoc power analysis using the observed standard deviations and treatment means revealed that, while the power to detect small differences between adjacent concentrations was moderate (β = 0.34), the design was sufficient to detect the statistically significant stiffness increase observed at the 4% ZnO treatment (p = 0.02). The sample size was selected to balance practical constraints with alignment to published methodologies and to ensure consistent replication across independently prepared samples. Future studies may explore increased replication or stratified sampling to further refine sensitivity for detecting small mechanical effects.

Results and Discussion

An essential element of the project was to devise and validate a laboratory-scale technique that allowed for (a) the even distribution of ZnO particles within the polymer matrix, (b) the shaping of the ZnO-enhanced resin into predetermined molded forms, and (c) the production of cured samples devoid of major air inclusions. Planetary mixing was utilized to meet these goals, as detailed in Figure 1. Additionally, a critical step in this methodology includes the manual injection of the mixture using standard catheter tip syringes from the bottom of the mold and conducting the curing process under pressure to further ensure the integrity and uniformity of the final product. The color of the three components of the mixture allowed for quick, accurate validation of zinc oxide dispersal within the cured plaques. The resin (white) and the catalyst hardener (deep blue) blended in a 10:1 ratio reveal a rich blue plaque when cured. Zinc oxide is a highly effective white pigment often used in paints to brighten whites. Therefore, when mixed into the polymer it lightens the blue color noticeably. A plaque with consistent color across the surface and cross-sectional areas demonstrates a process that achieved consistent blending of hardener and resin along with consistent dispersion of ZnO particles throughout the matrix. All plaques manufactured in the technique displayed consistent coloring throughout the polymer matrix and displayed no agglomeration of ZnO particles under 10X magnification.

Average Shore A Hardness was 48.59, 48.52, 47.71, and 47.99 for 0, 1, 2, and 4% treatments, respectively (Figure 4). Dunnett's test indicated the addition of ZnO at concentrations of 1%, 2%, and 4% did not statistically alter the silicon's hardness ($p > 0.18$) compared to 0% ZnO.

The average Young's Modulus for the prepared samples is shown in Figure 6. A Levene's Test showed the data to have statistically equal variance ($p = 0.05$), and a one-way ANOVA revealed a statistically significant difference in treatment means at a 0.05 confidence level. Therefore, a Dunnett's test was performed to compare the Average Young's Modulus for the 1%, 2%, and 4% treatments to the 0% treatment. Figure 5 illustrates the elastic progression of the material during testing, while Figure 6 presents the stress–strain curves generated from the same tests. At concentrations of 1% and 2%, ZnO did not significantly affect the silicon's stiffness, as indicated by the lack of statistical significance in the changes ($p > 0.96$). At a 4% concentration, the addition of ZnO did lead to a small, yet statistically significant, increase in stiffness, as evidenced by a p-value of 0.02, indicating a less than 5% chance that this result is due to random variation. The Average Young's Modulus (32.17) at 4% weight fraction indicates a 2% increase in stiffness from the control, demonstrating an effect of ZnO on enhancing the material's rigidity at this concentration.

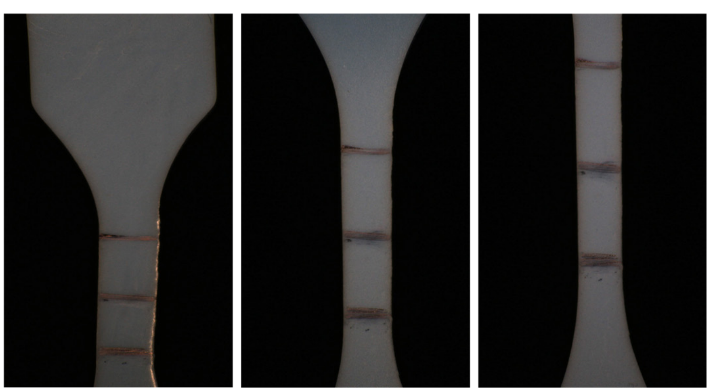

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Figure 5: Elastic progression of polymer in strain conditions of (a) 0%, (b) 25%, and (c) 50%.

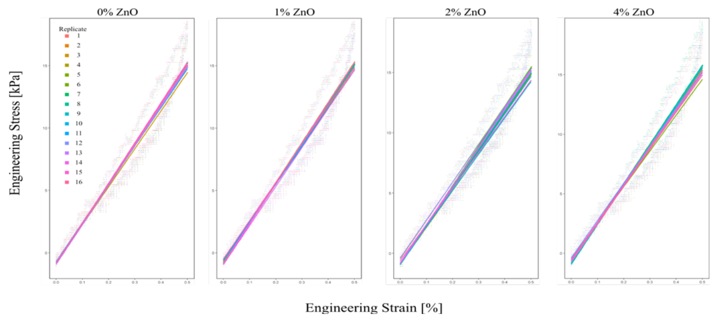

Figure 6: Average Young's Modulus by treatments of zinc oxide in silicone polymer matrix.

Conclusions

In this study, we explored the development of silicone resin composites by integrating platinum-cure silicone polymer (Smooth-Sil 950, Smooth-On, Inc., Macungie, PA, USA) with zinc oxide particles. The fabrication of these composite coupons utilized a pioneering technique designed to achieve uniform distribution of zinc oxide particles within the composite matrix. This approach addresses a critical aspect of composite material science: the precise positioning and concentration of additives to optimize material properties.

The mechanical properties of these composites, particularly their elastic moduli, were evaluated through uniaxial tensile testing and compared against various theoretical models cited in the literature. This comparison serves not only to validate the experimental approach but also to position the findings within the broader context of materials science research.

A significant aspect of this work lies in its focus on the mechanical characterization of the fabricated composites by varying the zinc oxide content. Zinc oxide is widely recognized for its antimicrobial properties, which have been substantiated through numerous studies. These properties have been demonstrated to provide antimicrobial action against pathogens directly associated with dairy mastitis when embedded within silicon polymers [16] and shown effective in limited biofouling on polymers in marine environments [17]. We sought to investigate the impact of ZnO additive on the mechanical properties of silicon polymers. The results indicate fabricated samples exhibited mechanical characteristics akin to those of dairy milking liner materials as documented in existing literature, and the hardness and elasticity of the polymer matrix was not negatively impacted by the addition of ZnO particles at concentrations under 4% *w/w*. These results are supported by Englert, et al. who found that silicon rubber matrices can withstand up to 10% addition of fillers without impacting hardness (2022).

Adding ZnO to the silicon did not significantly change the hardness of the material at any of the tested concentrations (1%, 2%, and 4%). This is indicated by high p-values ($p > 0.92$, $p > 0.18$, and $p > 0.22$), suggesting that any differences observed in hardness with these ZnO additions are likely due to random variation rather than a real effect of the ZnO. Similarly, adding 1% and 2% ZnO did not significantly affect the stiffness of the silicon, as the p-values are high ($p > 0.82$ and $p > 0.64$). However, at a 4% concentration, ZnO caused a slight but statistically significant increase in the stiffness (Young's Modulus) of the material, as indicated by a p-value of 0.01. The material became 2% stiffer, suggesting that the ZnO addition at this level has a real effect on increasing the stiffness of the silicon formulation. In summary, while the addition of ZnO up to 2% concentration didn't significantly impact the hardness or stiffness of the silicon material, a 4% concentration did make the material slightly stiffer, which is statistically significant and not likely due to chance. In previous work, Allen et al. [16] demonstrated the antimicrobial efficacy of ZnO-infused silicone polymers against *Staphylococcus aureus* strains associated with dairy mastitis, reporting a 74% reduction at 2% ZnO and a 99.5% reduction at 4% ZnO. When combined with the mechanical testing results from this study, these findings suggest that a 2% ZnO concentration may offer antimicrobial benefits without compromising the hardness or elasticity of the polymer. Additional work that illuminates impact to elongation at break, impact resistance, and flexibility would further support implementation of ZnO as a functional additive to silicon polymers in the dairy industry.

Incorporating functional additives such as zinc oxide into silicone resins offers a strategic pathway for enhancing the performance and applicability of these materials in dairy industry settings. The ability to control both the distribution and concentration of zinc oxide within the silicone matrix enables targeted optimization of composite properties. This approach not only advances materials science but also holds significant practical value in the design of dairy equipment, where antimicrobial efficacy and mechanical durability of contact surfaces are critical and directly impact the economics of dairy operations.

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