SpoilerAlert!: Final Report

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CHE 489: Team 1

Adam Bogart
Ryan Branch
Joanna Ciatti
Michael Conder
Madison Corradi

April 20, 2018

SpoilerAlert!: In-the-Cap Milk Spoilage Detector
Final Report

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Adam Bogart, *Marketing Director*
Ryan Branch, *Science and Innovation Director*
Joanna Ciatti, *Quality Assurance Director*
Michael Conder, *Regulations and Safety Director*
Madison Corradi, *Communications Director*

Prof. Erdogan Gulari, *Technical Supervisor*
Dr. Elaine Wisniewski, *Communications Supervisor*

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EXECUTIVE SUMMARY

In 2010, Americans consumed, on average, 20.4 gallons of milk per person.[1] Dairy milk is an everyday consumer item being bought and sold in large quantities daily across the United States. However, it is also a perishable product, and typically features only blunt measures of spoilage detection, such as sell-by or use-by dates. These dates are assigned relatively arbitrarily and bear no real significance on whether the product has gone bad, leading the consumer to potentially waste good product, or consume product of poor quality.

The SpoilSquad! conducted market research to determine if there is a market for a better milk-spoilage detection device, and if so, what the consumer requires in order for that device to satisfy their needs. The survey received nearly 350 responses and indicated to the SpoilSquad! that a market exists for a device that can accurately detect milk spoilage. In fact, consumers were willing to tolerate, on average, a price increase of $0.25 per milk purchase for a container that included a functioning detection technology. Upon interviewing milk producer Washtenaw Dairy, SpoilSquad! found that *SpoilerAlert!* is a device that milk producers would also be interested in implementing in their containers.

The SpoilSquad! proposes the *SpoilerAlert!* technology as a solution to the ambiguous issue of determining when dairy milk has spoiled. *SpoilerAlert!* is a food-safe, in the cap, CO_2-sensitive indicator that changes from pink to white when milk has spoiled. It provides peace of mind to parents and college students alike, that the product they are consuming or feeding to their children is still of good quality.

To ensure that *SpoilerAlert!* provides the best possible technology to all consumers, the engineering team has designed a series of experiments to systematically determine operating parameters. Additional rigorous functionality and quality assurance tests have been proposed, and *SpoilerAlert!* will be required to pass them before moving on to the consumer. *SpoilerAlert!* has been designed to endure any stressors, whether they be chemical, mechanical or otherwise, that it could reasonably be exposed to throughout the product lifetime.

Upon completion of product prototyping experiments, the SpoilSquad! will begin marketing the product to consumers. The engineering team hopes to implement *SpoilerAlert!* at the bottling phase of milk production, with producers purchasing and selling milk at a slightly higher cost, which would ultimately lead to consumers absorbing the cost of *SpoilerAlert!* with a milk price raise of no more than $0.25 per gallon. The goal of *SpoilerAlert!* is to cut down on consumption of spoiled milk and reduce the waste of good-quality product in the lives of milk consumers everywhere.

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NOMENCLATURE TABLE

Abbreviation	Full Name
Aq	Aqueous
CO_2	Carbon Dioxide
DI	Deionized
EtOH	Ethanol
FDA	Food and Drug Administration
KOH	Potassium Hydroxide
LbL	Layer-by-Layer
mg	Milligrams
mL	Milliliters
uL	Microliters
% v/v	Percent by volume

PRODUCT DEVELOPMENT

Following the market research and product development plan set forth last semester, the SpoilSquad! began development of *SpoilerAlert!* The following sections detail the journey from idea to prototype beginning with where we left of last semester.

I. INTRODUCTION

The University of Michigan's undergraduate Chemical Engineering curriculum culminates with a senior design project, in which engineers put their design and problem-solving skills to the test. In ChE 488, our team was assigned to recognizing a need, proposing a solution, identifying the market, and designing a product to solve the problem or opportunity. The SpoilSquad!, has developed a spoilage detection sticker to be used inside the caps of dairy milk. This report summarizes the ambiguity of milk spoilage and its detection methods, assesses the market opportunity for a solution, and details the development of *SpoilerAlert!*.

II. PROBLEM AND OPPORTUNITY STATEMENT

Milk exists inside nearly every refrigerator in the country. However, as milk is a perishable food, it spoils over time creating a sour taste and an undesirable consistency. Based on news articles and academic papers, it appears that there are very few commercially available products for detecting food spoilage. One of the most promising technologies in development of food spoilage devices are known as "Smart Tags" and are a nanorod-based solution which change color over time to indicate food expiration. However, the tags don't respond to chemical changes in the food but instead act as time-temperature trackers which provide a more accurate measure of countdown to expiration than the typical system of labeling food with a "best by" or "sell by" date. There is still no spoilage detection method that responds to the actual spoiling of milk. A consumer good as popular as milk requires an effective spoilage detection method.

Based on reviews of similar technologies to ours, we believe there is a great market opportunity for an in-the-cap spoilage detector. Concerns in food safety are paramount for many consumers. When *SpoilerAlert!* sent out a survey to gauge the markets' dairy milk consumption habits, 85% of respondents admitted to experiencing a milk spoilage issue. Customers are willing to tolerate a price increase in order to ensure a reliable detection method for their milk. Commercially, milk suppliers are willing to implement a technology such as ours to gain a competitive advantage. For an industry as big as the dairy milk industry, a product to ensure safe milk has potential for cost margins to multiply into a successful business while promoting food safety for all consumers who use it.

III. MARKET RESEARCH FINDINGS

To gauge the market for consumers who would ultimately purchase our product, *SpoilerAlert!* conducted market research, which included a Qualtrics survey of consumers and an interview with a milk seller, Washtenaw Dairy.

III.1 Qualtrics Survey

The survey of consumers was conducted using Qualtrics survey software (Qualtrics, Provo, UT). We released our survey to friends and family, and the general public by way of social media and in total, we received 350 responses. We asked questions that would give us insight into the milk market, the need for a detection method, and the price elasticity of this type of technology. These questions can be found in Appendix A. In addition, our questions featured a demographics section that would highlight the specific groups of people that our product would be marketed towards. Our complete survey responses are available in Appendix B, and responses to select questions are discussed below. The survey confirmed our hypothesis that there is no clear method for the detection of milk spoilage. In addition, the demographics section of our survey revealed insight into the types of consumers that our product will be catered towards, namely parents and college students. We first wanted to gauge the frequency in which our consumers buy dairy milk. Figure 3.1 below highlights the responses to the question:

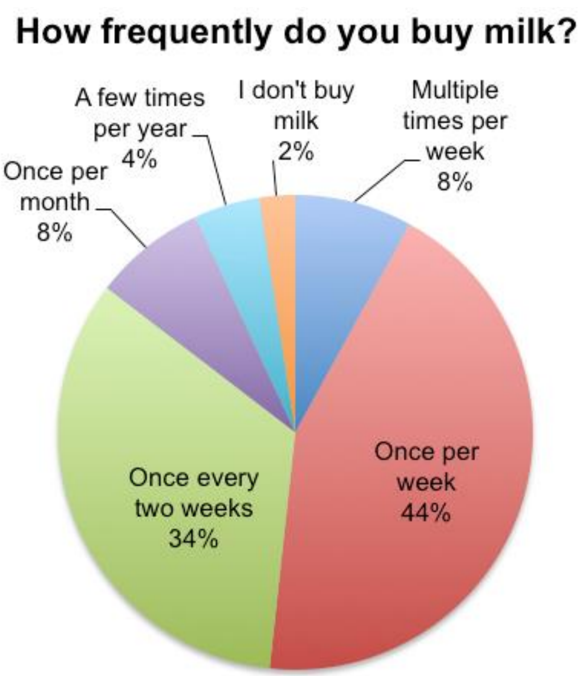


Figure 3.1. Survey data regarding frequency of milk purchasing.

The responses to this question feature an overwhelming majority of people purchasing milk once every week or once every two weeks. The frequency with which consumers buy milk indicates to us that the majority of respondents typically have milk in their refrigerator. However, the question also gives us insight into how long milk tends to last our consumers. Because milk tends to last roughly a week or two past the sell-by date, we expect our respondents who buy milk once every couple of weeks to run into spoilage at the end of their milk's usage. These results were a positive sign in confirming our hypothesis for a need of accurate spoilage detection.

To assess the demand for our detection method, we asked consumers how much they would be willing to spend on this type of technology. Our product could be fully functional, but *SpoilerAlert!* will be considered unsuccessful unless we are able to operate at a profit. Therefore, it would be necessary for consumers to pay extra for the product, enough to cover the cost of its implementation. Figure 3.2 breaks down the extra cost (in cents) that respondents would be willing to pay for milk-spoilage technology.

A breakdown of the data shows that on average, respondents would be willing to pay an extra 25 cents for milk containing *SpoilerAlert!* technology, and some people would pay at least one dollar. However, we will lose a lot of consumers if the price exceeds the 20-30 cents range and again in the 50 cents range. As such, our team has set a goal of charging only 25 cents extra per milk container for *SpoilerAlert!*. As 25 cents is our target price, we will work towards a variable operating cost below 25 cents for the production of *SpoilerAlert!*.

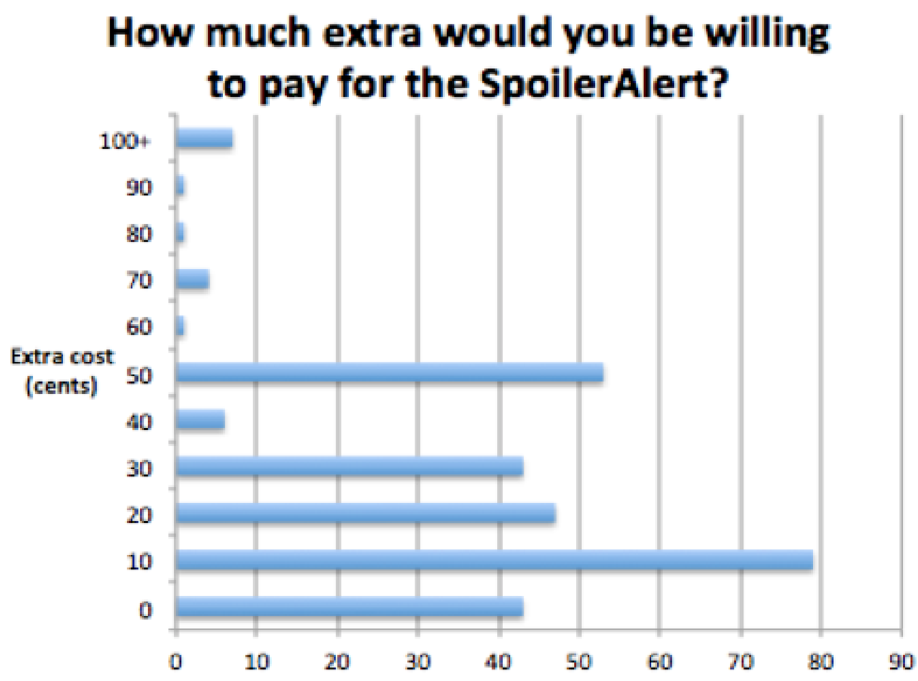


Figure 3.2. Survey data regarding tolerable cost of *SpoilerAlert!*

An important question in determining the implementation of *SpoilerAlert!* is fitting the product to a certain sized container or cap. As differing quantities of milk can have different sized caps, we asked consumers what quantities of milk they typically buy. Their responses are shown below in Figure 3.3.

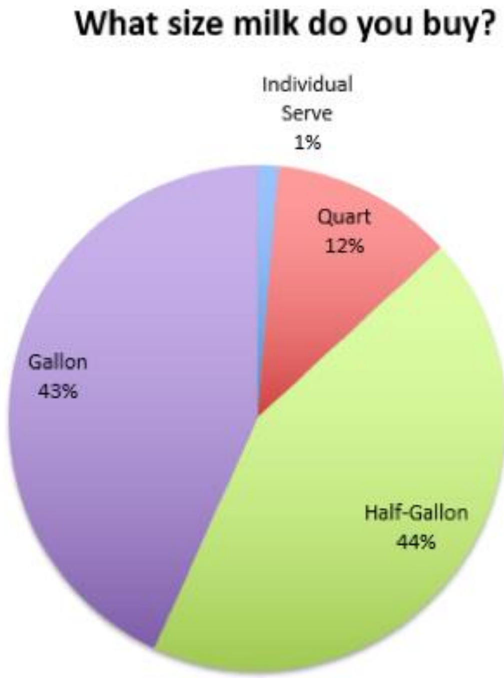


Figure 3.3. Survey data regarding milk quantities purchased by consumers.

The gallon and half-gallon sizes have shown to be the overwhelming majority of all sizes bought by our consumers, which is a positive result for our team because gallon and half-gallon containers often share the same cap design. As a result, we will cater our product to fit inside the cap of a gallon or half-gallon container. Another consideration our team takes seriously is whether consumers would have food safety concerns regarding their milk coming into contact with our new sticker technology. However, this fear was underrepresented in our survey results, as shown in Figure 3.4; we found that not only did the vast majority of consumers feel unconcerned, but that the concern over food safety seemed to lessen with the hypothetical addition of our technology. Respondents said that ensuring the freshness of their milk was a greater concern than the technology making contact with their milk.

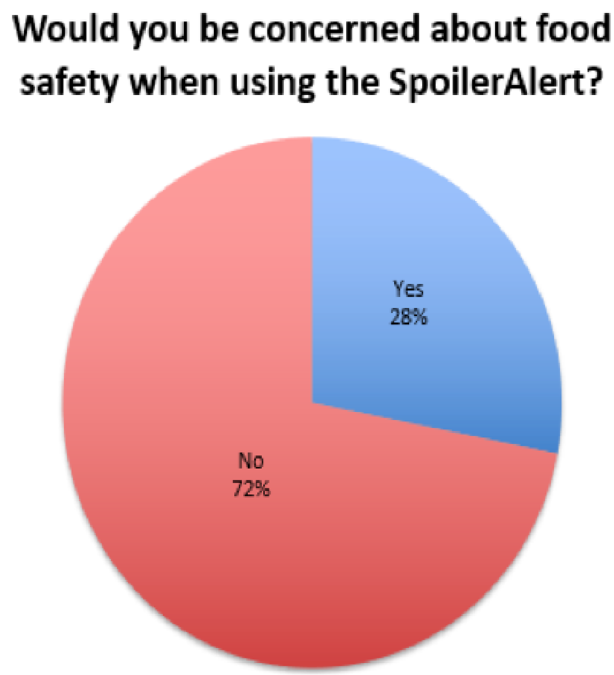


Figure 3.4. Survey data regarding *SpoilerAlert!* food safety.

III.2 Washtenaw Dairy Interview

The next part of our market research is analyzing the other members of the milk production line. As interested as consumers may be in the product, milk suppliers need to be willing to implement the technology in order for *SpoilerAlert!* to reach its potential. The product will be best distributed and utilized if bottlers agree to integrate *SpoilerAlert!* while bottling the milk. Therefore, we need milk suppliers and bottlers to work with us to implement the sticker. We met with the milk supplier for a substantial part of Ann Arbor, Washtenaw Dairy. Mary Jean Raab, President and Board Chair at Washtenaw Dairy, was able to provide a unique perspective on the life of dairy milk before the shelf at a grocery store. From Washtenaw Dairy's perspective, most consumers are forced to use the sell-by date in order to determine if their milk has spoiled, which is an inaccurate way of determining spoilage. From the store to any exposure post-purchase, sell-by dates are not indicative of the conditions that the milk experiences after it leaves the bottler. For this reason, we were told that *SpoilerAlert!* would be an asset that Washtenaw Dairy would certainly be interested in implementing. More detailed notes from the interview are included in Appendix C.

III.3 Market Research Analysis

The market research our team conducted gave us great insight into what demographics to target within our consumer base, in addition to supplying important information for consideration during the formulation of our *SpoilerAlert!* product. We were able to confirm the high volume of dairy milk purchases, gauge the amount of spoilage that our consumers face, and determine cost factors for the target market for *SpoilerAlert!* We have determined that *SpoilerAlert!* will effectively address the need for a reliable and accurate method of spoilage detection, and we will work to deliver a product that fills the aforementioned market void.

IV. TARGET MARKET STATEMENT

Our survey revealed that a milk spoilage detector would be highly utilized and purchased by a variety of consumers, however it clearly highlighted two target groups. First, parents who buy milk for their children. Two out of three respondents with children already pay extra for dairy milk features, such as chocolate milk, organic milk, etc. Within the parental demographic, 81% are willing to pay at least 10 cents for a spoilage detecting technology. Another demographic with a great need for *SpoilerAlert!* is the college-aged student (18-24), who may buy milk for themselves and their roommates as well. Busy college students reported to buying milk less frequently than their older counterparts, which led to one-third of respondents aged 18-24 to experience milk spoilage at least half the time. In addition, a quarter of these college-aged respondents admitted to drinking bad milk before realizing it had spoiled. The need for a detector within students will drive *SpoilerAlert!* sales, while price inelasticity of parents buying milk will drive sales for food safety as well.

V. CONSUMER NEEDS AND PRODUCT FEATURES

In our market research survey, our consumers indicated the features that were most important to them included being: cost-effective, food-safe, reliable, accurate, easily identifiable, and odorless. An overview of our needs and features can be seen in Table 5.1.

V.1 Essential Features

To ensure the safety of our product, we will be following all relevant Federal Drug Administration (FDA) guidelines. Our product will consist only of materials pre-approved by the FDA to be safe for our purposes. This feature is essential to *SpoilerAlert!* as it cannot be sold commercially if it is unsafe for food contact.

To make our product accurate and reliable, we plan on recording any false positives and negatives to ensure such mistakes do not have a statistically significant impact on the detection of spoiled milk; our product cannot consistently activate before or after the milk has spoiled. This feature is essential, as our product serves no purpose if it cannot reliably tell the consumer each time when their milk has spoiled.

V.2 Desirable Features

In order to make our product cost-effective, we are aiming for a price per sticker of $0.25, which is the average price that consumers said they would be willing to pay for our product in our market research survey, and further confirmed as a reasonable price by Washtenaw Dairy. The need to maintain a price of one quarter is labeled as desirable, as we ideally would like to price *SpoilerAlert!* Such that we would maximize profit while still producing a functional product.

V.3 Useful Features

Our product undergoes a very distinct and noticeable color change upon milk spoilage, allowing us to satisfy the easily identifiable criteria set forth by our consumers. The respondents to our market research survey indicated a desire for a more obvious sign that their milk had spoiled aside from an in-the-cap style detector. While our team attempted to implement feedback, due to necessary design changes we were unable to achieve more obvious clarity, and with it the ability to identify spoilage without opening the milk jug. However, these features are designated as useful because they were requested by consumers and add to the ease-of-use of our design, but are not absolutely necessary to the overall functionality. An overview of the consumer needs and features can be found below in Table 5.1.

Table 5.1. Consumer needs and product features breakdown.

Consumer Needs	Product Features	
Food-safe	Made of FDA-approved materials	✓
Accurately tests for milk spoilage	Activates at specific CO_2 level	✓
Cost-effective	Price per sticker under $0.25	✓
Easily identifiable	Recognizable color change	✓
	Can be identified from outside of jug	✗
No spoiled milk smell	Can be identified without opening	✗

VI. LAB NARRATIVE

The following sections describe the experimentation, challenges and new developments involved in the end production of *SpoilerAlert!*

VI.1 pH Experiments

The original concept for *SpoilerAlert!* was a pH-responsive layer-by-layer (LbL) film that would dissolve at a designated "spoiled" pH in the milk, thus allowing for food dye contained under the film to contact the milk and change its color. Thus, our team spent the first few weeks of the semester investigating the effect of milk spoilage on the pH of milk. We conducted several experiments to gain a full understanding of the milk spoilage process. Our team carried out experiments with skim, 2% and whole milk at both room and refrigerated temperatures. Additionally, we conducted experiments where milk was removed and replaced after pH testing, and where the milk was not replaced to allow us to observe the effect of increased headspace. However, all of these experiments returned largely the same results: the pH of our milk was not changing as it spoiled, as we had expected based on previous literature. Further research suggested that a potential reason for little pH change is that the milk we regularly buy is buffered. Upon receiving these results, we came to the realization that we would not be able to pursue our LbL film any longer because it was entirely dependent on the change in pH, so as suggested by Professor Gulari, our team elected to switch to a CO_2-based detection system.

VI.2 Adhesive Experiments

While waiting for our CO_2 meter to arrive so that we could begin tests for our new design, our team worked toward establishing other aspects of our product starting with the adhesive used to affix the sticker to the inside of the milk cap. The adhesive we selected would need to meet certain FDA guidelines, as described in the Standards and Regulations section of this report. FDA guidelines narrowed our search considerably and after researching possible food safe adhesives, our group agreed upon the FDA approved MAX CLR Epoxy Resin Adhesive.

Our selected adhesive has a designated FDA control experiment, which our team conducted twice with positive results, confirming its food safety and usability with our product. The experiment and its results are discussed further in the "Standards and Regulations" section of this report. However, beyond just food safety, our adhesive had to meet several other quality control criteria. Our adhesive cannot degrade when submerged or subjected to fresh or spoiled milk. Additionally, our adhesive must hold up mechanically during transportation and stocking of milk products. Testing adhesive integrity while submerged was straightforward, as we just attached a sticker to a cap and submerged it in water for approximately 10 days. At the end of the experiment, the sticker was still firmly secured to the inside of the milk cap. To test the integrity of the adhesive under chemical distress (spoiled milk), we attached stickers to the inside of four caps and placed them facing upward in four separate Petri dishes. Fresh milk was poured into each cap, such that it completely submerged the sticker. Two Petri dishes were left on the bench top, and two placed back in the refrigerator. The milk was left for two weeks and then carefully cleaned out of all four caps. At the end of the experiment, all four stickers were still secured to the milk caps, thus establishing that the adhesive holds up under any chemical degradation it is subjected to throughout the milk spoilage process. Finally, our team needed to ensure that our product would not experience mechanical failure. To test mechanical strength, we strove to find new and interesting ways in which mechanical failure may occur, including but not limited to: throwing milk caps at the wall and slamming milk caps on the table. While our adhesive was for the most part able to tolerate a great deal of mechanical stress, the one shortcoming of our adhesive is that it was unable to manage bending stress. If our cap was bent, the entire piece of cured adhesive would pop out. Our team was unable to carry out further adhesive experimentation because at the time of this revelation, we were focused on troubleshooting a more central component of our product, however if *SpoilerAlert!* were to be further pursued, it is recommended that more research and experimentation be done on adhesives.

Our final consideration for adhesives was regarding how much adhesive to use in the cap. We began by largely overestimating, so much so that our filter paper sank to the bottom of the adhesive and became useless for detection. We slowly decreased the amount in use until it was just enough to firmly attach our sticker. The selected amount to achieve desired attachment was 50mg.

VI.3 Sticker Pocket Fabrication

In addition to using our waiting time on adhesive selections, our group spent time focused on one of the most important aspects of our project: the pocket that houses our color changing indicator. As previously mentioned, our product has to meet certain FDA standards, and a substantial portion of determining what standards must be met by which components is determined by the permeability of our pocket housing. Obviously, our sticker pocket must be permeable to CO_2 such that it can dissolve into the indicator solution and change the color, however, our pocket must be impermeable to everything contained inside it: potassium hydroxide (KOH), ethanol (EtOH), and phenolphthalein. As the pocket chemicals are not food-safe components, it is extremely important that they are unable to cross through the membrane into the milk. Beyond the permeability criteria presented above, our pocket also needs to maintain its structural integrity and have the ability to be sealed.

Our team initially tapped Saran wrap as our desired pocket housing material. However, upon experimentation it became apparent that Saran wrap would not suffice for several reasons. First and foremost, it is too thin of a membrane, and too flimsy to maintain its shape. Additionally, it is extremely difficult to seal into a closed pocket, particularly because our chosen method of sealing is heat sealing.

After moving on from Saran wrap, our team discovered that Parafilm was a good candidate for our desired criteria. We began to evaluate the possibility Parafilm's implementation by attempting to determine a heat sealing procedure that could seal a piece of filter paper in between two Parafilm surfaces, producing a sticker, without tearing the Parafilm. The process took a bit of fine tuning, but we were able to produce filter paper pockets by heating a circular pipe fitting on a hot plate and stamping the filter paper between the two pieces of Parafilm, prior to removing the Parafilm paper layer.

Upon achieving a successful heat seal, our team proceeded to ensure that Parafilm is permeable to CO_2. At the time of the permeability experiment, our team was still working with the indicator bromocresol green, so we created a Parafilm pocket containing a piece of filter paper coated in bromocresol green indicator. We then exposed it to a stream of highly concentrated CO_2. Under these conditions, bromocresol green should undergo a blue (the color it turns when dripped on filter paper) to yellow color change, which is exactly what our team observed. The experiment confirmed the CO_2 permeability of Parafilm.

The final permeability criterion that Parafilm had to meet involved not allowing any of the liquids contained in our pocket to pass through, as well as being impermeable to ethanol vapor. In order to test the permeability of our liquids through the Parafilm we conducted two different experiments: one submerged and one inverted. In the submerged experiment, we created two complete pockets and submerged them, one under water and one under milk, and watched for a color change in the milk. If our indicator had been able to pass through the pocket, the milk would've changed color. However, because KOH and EtOH are clear substances that could've precipitated out of the indicator solution, we also performed a suspended test, in which our indicator solution was contained in a Parafilmed vial and inverted over an empty beaker to see if any solution was able to pass through. As both of these tests yielded an impermeable result, our team selected Parafilm as our sticker pocket material.

VI.4 CO_2 Quantification and Experimentation

After our CO_2 meter arrived, our team began to quantify the amount of CO_2 given off by milk as it spoils in order to appropriately calibrate our sticker to change colors when milk is spoiled. The primary experiment that we ran to obtain the data was to pour fresh milk into a Nalgene bottle and insert the CO_2 detector (effectively sealing the container). We then collected CO_2 data over the course of 48 hours (approximately how long it takes for full spoilage to occur based off previous experiments). We performed the experiment multiple times, slightly varying the procedure each time. The first iteration of the procedure involved pouring the milk into the container, inserting the detector and leaving it to sit for 48 hours, collecting qualitative data (how the milk looked) every 12 hours. After completing this experiment, we performed an experiment in which the set up was identical, but when we took qualitative data every 12 hours, we removed some milk from the container. Milk removal allowed us to take qualitative data about smell in addition to sight and modeled a more realistic scenario in which someone would open their milk and pour some out to drink it, enabling us to look at how the frequency of opening the milk and the changing headspace affected the rate of spoilage. The result of studying these effects was the realization that the instantaneous amount of CO_2 in the container wasn't important for our purposes, but rather the amount of CO_2 the sticker would accumulate would be the most relevant.

VI.5 Indicator Selection

Once our team had preliminary CO_2 data, we began testing with various indicators. Selecting an indicator was contingent upon the indicator passing a progression of color change tests. We began with concentrated CO_2 direct exposure tests, then concentrated CO_2 desiccator tests, dilute CO_2 desiccator tests and finally testing color change in the milk container itself. The indicator our team was initially working with was bromocresol green. While the bromocresol green stickers passed the concentrated CO_2 direct exposure test, they only showed slight color change around the edges in the concentrated CO_2 desiccator test, indicating to us that the amount of CO_2 milk would produce while spoiling would not be sufficient to cause color change.

After failure of the bromocresol green indicator, our group performed further research and calculations and found that methyl red indicator was a potential candidate for the pH range that we were working with. Upon receiving the methyl red indicator, our group first noticed that it is insoluble in water, so some amount of ethanol must be used to bring the methyl red into solution. After achieving desired dissolution of methyl red in solution, we prepared stickers. The methyl red stickers again passed the direct exposure test, but only demonstrated minimal color change in the concentrated CO_2 desiccator test, again leading us to the conclusion that the amount of CO_2 released in milk spoilage would be insufficient to change the color of our sticker.

After neither indicator worked, our team sat down with Professor Gulari and he recommended that we attempt to use phenolphthalein with added base, so our team prepared phenolphthalein in EtOH (190 proof) solution, which was clear. From this stock solution, we removed a small volume and titrated KOH into it until we obtained a desirable color difference (dark pink). We used a small volume of solution to create stickers and subjected them to our chronology of tests; the phenolphthalein solution passed each of our tests. Once we had a promising indicator, we sought to determine the ideal volume to use in each sticker so that it coated the entire piece of filter paper and appeared dark enough in the sticker, without overflowing during the heat sealing process. The ideal volume was identified as 125μL.

VI.6 Phenolphthalein and KOH Solution

The final aspect of product construction that our team had to consider was the concentration of KOH in each sticker. We had to find a concentration at which the color change accurately represented when spoilage was occurring, while maintaining a dark enough sticker color to be able to discern a clear color change. Our group tested concentrations of KOH in phenolphthalein solution as low as 6 uL/mL and as high as 10 uL/mL. What we found is that below 8 uL/mL the color on the sticker is too light for an easily identifiable color change, and above 9 uL/mL the sticker would take much too long to change to clear to indicate spoilage. Having narrowed our options down to 8 or 9 uL/mL, we ran simulated experiments with each in the milk container and found that 9 uL/mL had a color change more congruent with the spoilage of milk, and had the added benefit of exhibiting a darker pink color initially, making the change easier to identify. For these reasons, our group elected to use the 9 uL/mL KOH in phenolphthalein solution concentration.

FINAL PRODUCT

Following three months of work in the laboratory, the final, working prototype of *SpoilerAlert!* was fabricated. The technology involved in the successful product is not the same as the planned layer-by-layer film technology, but instead operates using a pH indicator solution that changes color upon exposure to a certain amount of carbon dioxide (CO_2) gas.

VII. FINAL PROTOTYPE

The final *SpoilerAlert!* sticker prototype is a semi-permeable membrane made of Parafilm M that encases a Whatman filter paper (25mm diameter) which has been soaked in an indicator solution. The prototype is a flat, circular disk approximately 3 cm in diameter weighing about 10mg. A photo of the finished *SpoilerAlert!* sticker, before and after being attached to a cap is shown in Figure 7.1, below.

(a) **(b)**

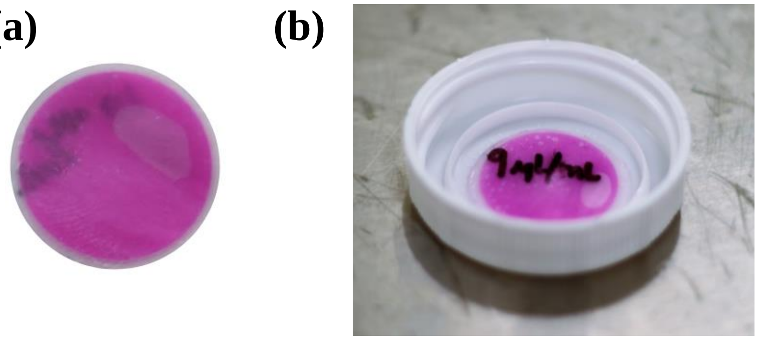


Figure 7.1 (a,b). (a) *SpoilerAlert!* sticker before being placed into a cap, background removed from image for clarity. (b) *SpoilerAlert!* in use as part of a milk jug cap, after being adhered to the cap underside using FDA-approved food safe epoxy MAX CLR.

VII.1 How It Works

The prototype functions based on microbiology and simple acid/base chemistry. The lactic acid bacteria that are naturally present in milk are constantly metabolizing lactose, a sugar present in milk, and excreting lactic acid and CO_2, see Figure 7.2.

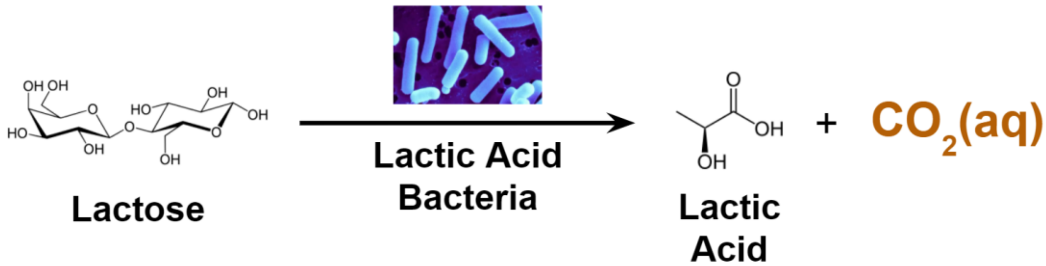
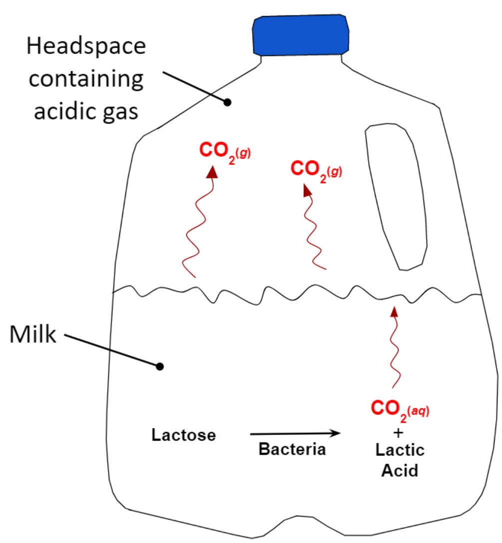


Figure 7.2. Lactic Acid Bacteria metabolize lactose (sugar) into lactic acid and aqueous carbon dioxide in milk.

As lactic acid is produced, the proteins in milk, especially casein, begin to denature and separate from the rest of the milk solution, eventually forming coagulated chunks in the milk which is what most people think of when they hear "spoiled milk". However, when milk is kept in a refrigerator, the lactic acid bacteria grow much more slowly than at room temperature, so the milk stays fresh and free of chunks for weeks, rather than two days at room temperature. In tandem with the production of lactic acid and milk coagulation, is CO_2 release from the milk as it is produced by the lactic acid bacteria. The tiny CO_2 bubbles do not remain in the milk solution but rather leave the aqueous phase and reside in the headspace inside the milk container, above the milk, shown in Figure 7.3. Thus, the CO_2 concentration in the headspace above the milk increases over time, as milk spoils, and can be used as a metric for milk freshness, as in *SpoilerAlert!*.

Figure 7.3. Milk jug containing milk and releasing CO_2 as it spoils.

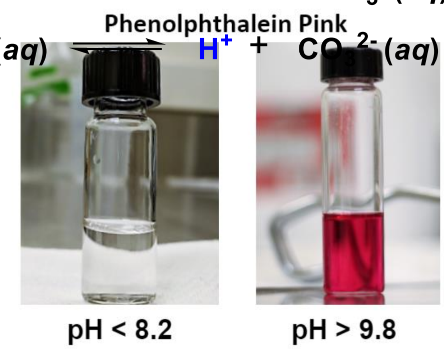


For our product to function correctly, a color change of the indicator solution must occur when milk changes from fresh to spoiled. A color change occurs when the CO_2 concentration above the milk exceeds our threshold that was determined with experiments where the CO_2 concentration was measured over time as milk spoiled at room temperature, and the milk condition was correlated with carbon dioxide concentration. As more CO_2 becomes present in the container, its partial pressure (P_{CO2}) increases, which shifts the concentration of CO_2 dissolved in the indicator solution of ethanol based on vapor-liquid equilibrium and Henry's Law constants. When the concentration of CO_2* increases in the solution, the pH decreases because CO_2 is an acidic gas that reacts with water to form H_2CO_3*, HCO_3^-, and CO_3^{2-} according to the equilibrium reactions 1, 2, and 3, below.

$$CO_2\,(g) + H_2O \rightleftharpoons H_2CO_3^*\,(aq) \qquad K_H = 10^{-2.2} \qquad (1)$$

$$H_2CO_3^*\,(aq) \rightleftharpoons H^+ + HCO_3^-\,(aq) \qquad pK_{a1} = 6.3 \qquad (2)$$

$$HCO_3^-\,(aq) \rightleftharpoons H^+ + CO_3^{2-}\,(aq) \qquad pK_{a2} = 10.3 \qquad (3)$$



The pH indicating dye that we are using is phenolphthalein which changes from fuchsia pink at pH above 9.8 to colorless at pH values below approximately 8.2 in ethanol (Figure 7.4).

Figure 7.4. Phenolphthalein pink indicator solution showing drastic color change from pH less than 8.2 (left) to pH greater than 9.8 (right).

The dissolution of CO_2 produced by the milk through the semipermeable membrane to the indicator solution is the cause of the color change, and allows us to reliably indicate to consumers when their milk is no longer drinkable. A cross-sectional schematic of *SpoilerAlert!* at pH 9.2 and below pH 8.2 is shown in Figure 7.5.

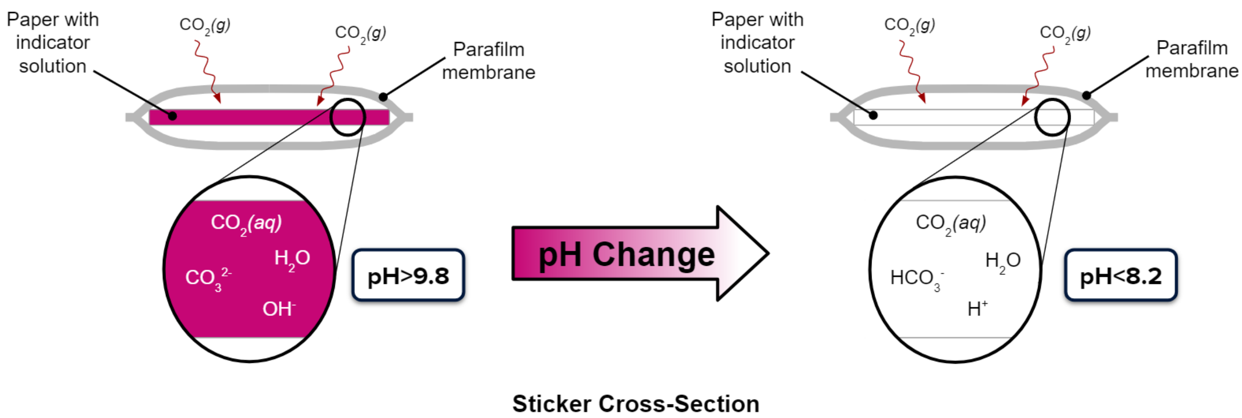


Figure 7.5. Cross-sectional schematic of *SpoilerAlert!* sticker at pH above 9.8 (left) and below 8.2 (right) which signals milk spoilage based on CO_2 concentration.

VIII. MATERIALS AND EQUIPMENT

The materials and equipment used during fabrication of the prototype are listed below in Table 8.1. Our *SpoilerAlert!* sticker is composed of six key materials.

Table 8.1. Materials and amounts required for the fabrication of a *SpoilerAlert!* sticker.

Material	Amount Required
MAX CLR Epoxy Resin Mixture	50 mg
Filter Paper	1 circle of 20mm diameter
Parafilm M	2 in^2 (approx. 12.9 cm^2)
Ethanol, 190 proof	125 µL
Potassium Hydroxide*	63.1 µg
Phenolphthalein*	8.75 mg

*Because the amounts of potassium hydroxide and phenolphthalein are so small, it is impractical to measure and prepare the amounts needed for a single sticker. Instead, the entire solution which is encased by Parafilm is produced in bulk, by creating a 70 gram per liter solution of phenolphthalein in 190 proof ethanol and adding 1 Molar KOH solution such that it is 0.9% v/v in the final mixture.

The mixture of ethanol, potassium hydroxide, and phenolphthalein serves as an indicator solution which has been titrated to an exact pH. The mixture has the property that the presence of CO_2 will cause it to lose all of its pink color at precisely the gaseous carbon dioxide concentration range that one would expect when milk has spoiled. The solution is absorbed into a circular piece of filter paper in order to provide structure and ensure that the liquid remains evenly distributed throughout the sticker. Parafilm M is heat-sealed around the filter paper to create a watertight "pocket" through which allows carbon dioxide to diffuse through while holding in the indicator solution ingredients. Finally, the MAX CLR epoxy resin mixture is a water-insoluble, food-safe adhesive compound which is used to adhere the Parafilm pocket onto a standard milk cap. Additionally, certain equipment is required to fabricate each SpoilerAlert product, listed in Table 8.2.

Table 8.2. Equipment directly involved in the creation of a *SpoilerAlert!* sticker.

Equipment	Role
Scissors	Cutting Parafilm roll down to proper size
Digital scale	Measuring phenolphthalein
Beaker, 100mL	Measuring ethanol and mixing indicator solution
Stir plate and stir bar	Mixing phenolphthalein into ethanol until dissolved
Micropipette, 2 – 20 µL	Measurement of KOH solution into phenolphthalein solution
Micropipette, 100 – 1000 µL	Measurement of final indicator solution onto filter paper
Brass pipe cap, 0.75 in diameter	Heat-sealing Parafilm around filter paper in a circular pattern
Hot plate	Bringing the brass pipe cap up to 75 degrees Celsius
Tweezers	Accurately placing the heated pipe cap onto Parafilm
Metal tongs	Applying pressure to the pipe cap in order to melt and seal Parafilm

Finally, certain equipment was used throughout the product development process, but unnecessary for construction of the final product. This equipment and its role is listed in Table 8.3.

Table 8.3. Materials and equipment used during product development.

Material/Equipment	Role
pH meter	Measurement of indicator solution pH during titration
Carbon dioxide tank, 100%	Determination of an indicator's ability to change color from CO_2
Carbon dioxide tank, 1% in air	Determination of color change properties at milk-level CO_2 values
Desiccator	Isolated environment in which to expose stickers to CO_2
Carbon dioxide detector	Measurement of CO_2 concentrations created by milk during spoilage
Nalgene bottle, 250mL	Containment of milk in airtight environment during CO_2 measurement
Computer with Vernier Graphical Analysis 4 software	Prolonged data collection and saving of CO_2 concentrations during milk spoilage measurement experiments
Camera	Recording of time-lapse photography to observe sticker color over time
Milk	Creation of spoilage environments for data collection and testing
Tape	Temporary adhesion of stickers to inside of Nalgene bottle

IX. FINAL FORMULATIONS

The final formulation of *SpoilerAlert!* involves three different components, which will be defined and described in this section: the indicator solution, the Potassium Hydroxide (strong base) solution, and the sticker implementation. Because specific amounts will depend on the batch size for a group of stickers, concentrations and proportions of components will be used in this section. The exact specifications and formulations of these three components are as follows:

IX.1 Indicator Solution

The indicator for *SpoilerAlert!* is a phenolphthalein in 190 proof ethanol solution. The expected solubility of phenolphthalein in ethanol as per literature is 71 grams/liter ethanol. To prep the indicator solution, as much phenolphthalein should be dissolved in ethanol as possible. 70-71 grams per liter of 190 proof ethanol should be mixed and stirred over time to ensure proper solubility. Light heat up to 50C can be applied to expedite the solubility process, though any heat above this amount could dissolve ethanol solvent. Once at least 70 grams of phenolphthalein per liter of 190 proof ethanol have been dissolved, the indicator solution is successfully prepared.

It is important to note that different mixtures of ethanol will have different phenolphthalein solubilities and initial pH values upon phenolphthalein dissolution. The procedure outlined in this report is specific only to 190 proof ethanol for guaranteed reproducibility of *SpoilerAlert!*

IX.2 Potassium Hydroxide (Strong Base) Solution

The strong base solution does not involve any preparation but requires a precisely 1.0M (1.0 N) solution of KOH. Proper safety precautions should be taken as KOH is hazardous. Using a precise 20-200 microliter pipette, 9 milliliters of 1M KOH should be added to each liter of indicator solution. If a different amount of indicator solution is prepared, precisely 9mL/L indicator should be added to the indicator solution. The addition of base should turn the new solution purple. Mix the vial to ensure homogenous coloration. The resultant purple solution should be dark enough to clearly change the color of a white filter paper. Confirmation of the color change means successful production of the *SpoilerAlert!* indicator solution.

IX.3 Sticker Implementation

The final procedure involves the implementation of the solution when creating *SpoilerAlert!* Sticker. A hot plate should be set to heat up a roughly 2.5cm brass fitting so that the brass piece is extremely hot on the circular base. First, a 2cm filter paper circle should be placed on top of a parafilm strip. The filter paper should be placed on one half so that the other half can be folded over to fully enclose it. Then, using a 20-2000 microliter pipette, 125 microliters of solution should be added to each *SpoilerAlert!* sticker. Then, by folding the parafilm over to completely cover the pocket, tongs are used to add the hot metal circle around the outside of the filter paper on top of the parafilm. The tongs should be used to apply pressure to get a full seal from the parafilm layers around the filter paper. After 10 seconds of pressure, the metal should be returned from the hot plate. Then, the outside of the pocket can be peeled to create a sealed, circular pocket.

Adhesive component MAX CLR Epoxy Resin components A and B are to be mixed at a 2:1 ratio by mass, respectively. Mixing can be done using the back of a disposable pipette for 5 minutes. Then, a measured 50mg of resin mixture is to be applied to the inside of a cap. Applying pressure for 10 seconds is the best way to successful adhere the pocket to the cap, and as per the instructions provided by MAX CLR the adhesive is allowed 48 hours to cure at room temperature.

X. QUALITY ASSURANCE

Multiple tests were performed to ensure that the product is of sufficient quality and will not fail during use by consumers. The experiments that were undertaken have been split up according to type of testing and failure mode that is being probed by the test.

X.1 Mechanical Failure

The experiments that were run for mechanical failure included throwing the milk jugs and caps against the walls to simulate stocking, removing and replacing the caps containing stickers repeatedly to check the integrity of the parafilm barrier and adhesive. Based on these tests, we concluded that the stress incurred by stocking would not damage *SpoilerAlert*! and that removing and replacing the cap would not interfere with proper function of the device. We also found that if the cap is bent out-of-plane, the adhesive and sticker will pop out of the cap as a solid disk.

X.2 Parafilm Permeability

Experiments run for parafilm permeability included testing for permeability to CO_2, ethanol, and water. Carbon dioxide permeability was probed by exposing sealed sticker pockets containing various pH indicators to a CO_2 stream, an atmosphere of concentrated CO_2, and an atmosphere of dilute CO_2. These experiments showed that the parafilm is permeable to CO_2 as the indicator changed color within 2 minutes of being exposed to a stream of concentrated CO_2 and over the course of an hour when exposed to an atmosphere of CO_2 (pure and 1% CO_2). Ethanol and water permeability were probed by sealing ethanol and water into separate test tubes with parafilm and inverting the tube to look for liquid permeating the membrane. The results of these experiments showed that, as expected, the parafilm was impermeable to ethanol and water.

X.3 Chemical Degradation

Experiments run for chemical degradation included testing for degradation of the parafilm barrier, seal, and food-safe adhesive when exposed to water, fresh milk, and spoiled milk. Degradation was tested by sealing filter papers coated in food dye inside of parafilm and submerging these pockets (in duplicate) in fresh and spoiled milk, as well as water. If the seal or parafilm were to fail, the liquid that the pocket is submerged in would turn the color of the dye (green) – figure X-6, and if the adhesive failed, the pocket with adhesive would detach from the cap. The results of these tests showed no dye release or adhesive failure after 2 weeks of exposure to both types of milk and water.

X.4 Spoilage Indication

Experiments to test the spoilage indication of *SpoilerAlert!* included exposing the device which contained indicator-soaked paper to pure and dilute CO_2. Pure CO_2 was sprayed in a stream over the sticker pocket and after about 10 minutes, the sticker had turned from completely pink to almost entirely white. This was evidence that the indication method for CO_2 is successful. The next tests run for spoilage indication included placing multiple prepared sticker pockets into a clear-top desiccator chamber and charging the chamber with both concentrated and dilute (1%) CO_2 where they changed color in 3-15 hours.

The real-world tests for spoilage indication were conducted using four separate half gallons of milk, and four independent stickers. One sticker was attached to the interior of each milk cap and two of the jugs, one-third of the way full of milk, were left on the countertop to spoil while the other two jugs were left in the refrigerator as fresh milk. The caps from the spoiled milk turned white when the milk spoiled and those left on the fresh milk in the refrigerator remained pink, this is shown in Figure X-6. The results of this experiment are evidence that *SpoilerAlert!* will indicate when milk has spoiled but will not provide a false indication on fresh milk.

PRODUCT MANUFACTURING

The following section describes key considerations in the manufacturing process of *SpoilerAlert!* technology.

XI. STANDARDS AND REGULATIONS

Although *SpoilerAlert!* is not itself a food, it is regulated by the United States Food and Drug Administration in that it is considered an “Indirect Food Additive”. The FDA defines this category as things which will “come into contact with food as a part of packaging, holding, or processing, but are not intended to be added directly to, become a component, or have a technical effect in or on the food”. Because *SpoilerAlert!* can contact milk within the jug during regular use but will not become a component of or have an effect on the milk, it falls into the "Indirect Food Additive" category[6].

The FDA also has guidelines for Food Contact Substances, also known as FCS. A FCS is defined as “any substance that is intended for use as a component of materials used in manufacturing, packing, packaging, transporting, or holding food if such use of the substance is not intended to have any technical effect in such food”[6]. The FCS category is further defined through the existence of Food Contact Materials (FCM) and Food Contact Articles (FCA). In the case of *SpoilerAlert!*, two of our six product components come into direct contact with milk: Parafilm and MAX CLR Epoxy Resin. Neither of these components are composed of just one chemical compound, so they fall outside of the definition of FCS. Instead, they’re considered FCMs, being a mixture of one of more FCS. A finished product formed out of one or more FCM is an FCA, which is the category into which *SpoilerAlert!* as a whole belongs.

To be legally sold on a commercial basis in the US, any component of the product which comes in contact with food must be directly approved for use by the FDA. The Code of Federal Regulations is a document made available by the Government Publishing Office which codifies the general and permanent rules put into place by the departments and agencies of the Federal

Government. The first chapter of the 21st title of that document includes a large list of rules from the Food and Drug Administration, and subchapter B governs any food intended for human consumption. Subchapter B contains the entirety of information needed for the approved use of each of our indirect food additives, specifically under part 175 which is defined as "Indirect Food Additives: Adhesives and Components of Coatings".

Within this part, Subpart C sections 250 and 300 cover the use of Parafilm M, which is approved under definitions of rules for synthetic paraffin and for polymeric coatings[4][5].

The use of MAX-CLR Epoxy Resin is a bit more complicated. Although all of the substances which make it up are directly approved under Subpart B section 105, any adhesive material within this section is approved subject to certain experimentation which must be carried out based on the nature of its use. Table 1 of this document (not pictured for brevity) defines milk as an "Oil-in-water emulsion dairy product" which defines it as a IV-B food type[3]. For any given food type, the experiments needed for approval are defined by the condition of that food's use[3]. Milk's use involves refrigerated storage, causing it to fall under condition F which necessitates an extractant experiment using water at 70 degrees Fahrenheit over a 48-hour period. The procedure for testing approved use of our MAX CLR adhesive for the conditions of milk's storage and usage is outlined below. We decided to carry out 3 identical trials simultaneously to be more confident in our results.

1. Prepare 3 milk caps with our selected amount of adhesive (50 mg) present in each and allowed to cure for a 48-hour period at room temperature as instructed by MAX CLR
2. Find 3 suitably sized containers and clean them thoroughly before allowing to dry completely
3. Label each container and weigh it, recording the mass of the dry empty container
4. Place one milk cap into each of the three containers
5. Fill each container with room-temperature deionized water to a height of approximately ¼ of an inch away from the top
6. Seal the containers and place them into a room-temperature environment for 48 hours
7. Uncap the containers and remove the milk caps from each, leaving the water inside
8. Place the containers on a hot plate and evaporate the water until only about 5% of the original water volume is still present in the container. Avoid rapid boiling at all costs so that liquid water doesn't splash out of any of the containers during the evaporation process
9. Leave the containers open to the air for another 72 hours or until all of the remaining water evaporates
10. Weigh each container and compare its final mass with the initial mass recorded in step 3

Figure 11.1. Photograph of containers during step 8 of FDA experimentation.

The difference in mass is caused by the accumulation of any extractives which have leeched out from the cured MAX CLR adhesive epoxy resin mixture into the water. Based on the smallest intended usage of *SpoilerAlert!* being for half-gallon containers of milk, the absolute maximum amount of adhesive extractives which can be allowed within FDA regulations is 25 mg[3]. The experiment was performed multiple times, and an increase in mass at or above 10 mg was not measured. However, we did occasionally take measurements that implied that the mass had gone down over time instead of increasing at all. After consulting with Dr. Barr about the equipment used to make these measurements, he expressed his opinion that the result was due to slight inconsistencies in the scale's ability to take extremely precise measurements. His recommendation was to proceed as if the experiments had been fully confirmed to pass regulations, despite a lack of ability to directly confirm it. One notable point is the fact that our total amount of adhesive used per sticker is only 50 mg, and so half of the entire adhesive mass would need to leech out into the water for the product to fail the FDA test. Still, if *SpoilerAlert!* is to be sold commercially in the future, we strongly recommend that similar tests be repeated in a more high-precision environment before going to market.

XII. ENVIRONMENTAL AND SAFETY CONCERNS

While *SpoilerAlert!* is not expected to have a large environmental impact, its most significant impact would be that it does not use recyclable materials. It's inability to be recycled is compounded by the fact that it is a one-time use product, which potentially leads to hundreds of millions of stickers needing to be made per year, if implemented nationally. The only material used that does not directly make it into the final *SpoilerAlert!* product is the leftover Parafilm M after sticker cutting. It would be worth looking into the feasibility of using the leftover Parafilm M for new stickers. The *SpoilerAlert!* sticker could also lead to the improper disposal of chemicals. A very small amount of phenolphthalein and potassium hydroxide are used in the sticker, and these chemicals are not typically disposed of with regular household waste. While a review published by the U.S. National Library of Medicine has found phenolphthalein to not be environmentally toxic, it tends to accumulate in soil by adsorbing to the surfaces of rocks and sediment[2].

There are also several safety concerns with the *SpoilerAlert!* technology. Even though many physical durability and chemical degradation tests have been completed for quality assurance purposes, it is still possible to physically break open a sticker with enough force. While tearing isn’t expected to happen under any normal conditions, in the possibility of breaking the sticker’s seal, the contents, and anything that it has come into contact with, should not be consumed. It is also possible that someone would consume spoiled milk due to a faulty sticker, and, while there is no biological process that can make you ill from spoiled milk, one could still feel sick or vomit from the smell and taste.

XIII. PROCESS

The goal of *SpoilerAlert!* Is to implement it during the manufacturing stage of milk jug and cap or during the bottling process. Implementing our technology at either of these stages would mean that *SpoilerAlert!* is available to everyone immediately after purchase of their milk, no further assembly required. To get to the ultimate stage though, the sticker itself must be made. This process is outlined visually in Figure 13.1.

The process begins with two separate components in parallel: the sticker itself and the adhesive. The sticker contents, ethanol, phenolphthalein, and potassium hydroxide (KOH), are separately sent to a mixer in their previously determined ratios: 70 g/L phenolphthalein powder in 190 proof ethanol and 9µg/mL 1M KOH solution in the phenolphthalein solution. In the pocket assembly stage, the mixture is then dropped onto untreated filter paper, which rests on a 1”x1” square of parafilm (with its paper packaging). After the mixture has been dropped on the filter paper, another equally-sized piece of parafilm will be placed on top of it. The resultant setup will be sent to heat sealing, where a 5/8-inch diameter brass pipe cap presses the two pieces of parafilm together, sealing the filter paper inside a parafilm pocket. It is important to note that the hot metal only comes into contact with the paper of parafilm, not the plastic component. The metal is to be kept around 70-80°C, in order to be hot enough to seal the parafilm but not too tight to burn the paper and damage the pocket. In the sticker cutting section, the paper would be peaked off the parafilm, and parafilm pocket would then be punched out of the remaining parafilm. The sticker is now fully ready, and just needs to be adhered to the cap. In the second of two parallel production lines, the MAX CLR Resin A and B get mixed in a 2:1 ratio, respectively. After mixing these resin components, *SpoilerAlert!* is finally ready to be fully assembled. 50mg of the adhesive mixture is to be placed on each milk cap, and then the cut parafilm pocket is to be placed on top. The adhesive will cure in 48 hours, and *SpoilerAlert!* will be ready to use.

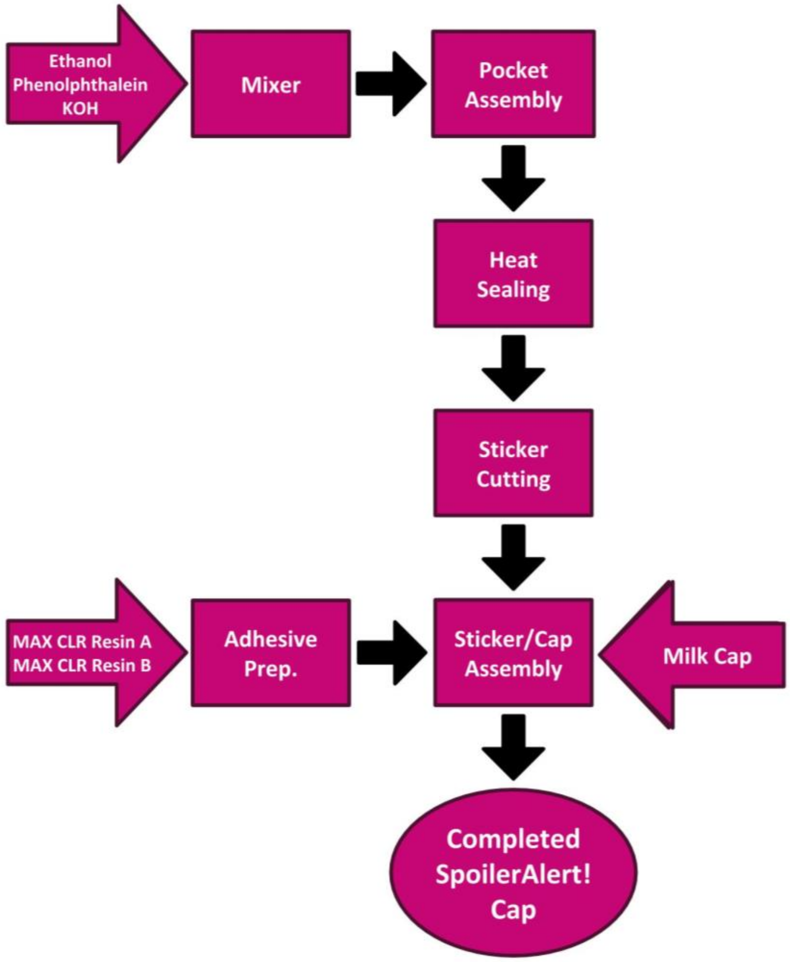


Figure 13.1 Process Flow Diagram for large-scale manufacturing of *SpoilerAlert!* Technology.

XIV. ECONOMIC ANALYSIS

Due to the low-cost nature of fixed costs associated with production of *SpoilerAlert!* relative to the production amounts, the variable costs of the raw materials will be the main components in determining the profitability. When determining the costs of all the components, we look at commercially available bulk values. Raw material costs are based off the biggest bulk values online and dividing the component size by the amount needed per sticker.

Table 14.1. Cost of each component for *SpoilerAlert!* at semi-bulk prices.

Material	Unit Cost
Filter Paper	1.0¢
Parafilm	0.5¢
Ethanol	0.48¢
Phenolphthalein	0.14¢
Epoxy	0.09¢
KOH	0.002¢
Total Cost:	**2.2¢**

In reality, mass production of *SpoilerAlert!* could be done in a much higher scale bulk working directly from distributors. An estimated 70% of the cost could be saved using a much higher scale bulk from a distributor. Therefore, the semi-bulk estimate of 2.2 cents brings the cost down to 0.66 cents, or two-thirds of a cent. From a commercialization and mass-production standpoint, this 0.66 cent estimate is a better estimate on the cost that SpoilSquad! would incur.

The other variable when determining profitability is the price that a sticker would cost. Pricing of *SpoilerAlert!* is based off market research and questions based off pricing. The following bar graph (Figure 14.1) represents the amounts 350 surveyed respondents are willing to pay.

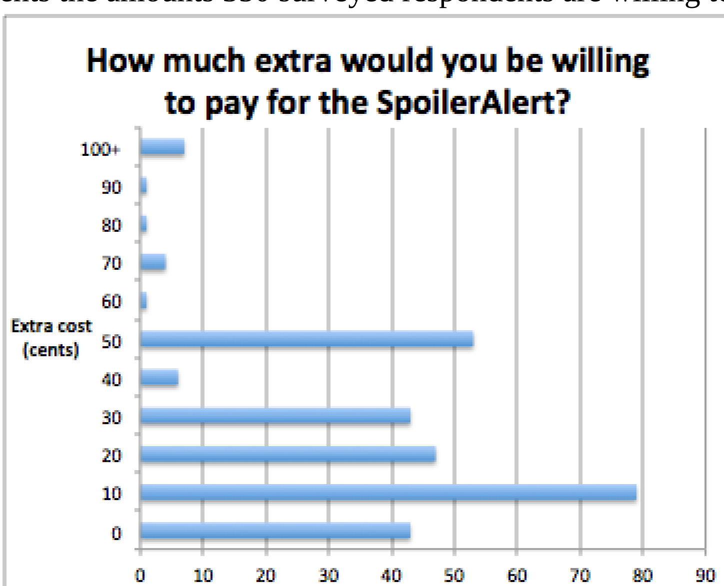


Figure 14.1. Willingness of survey respondents to pay a given price.

With information on price tolerance, we can create a demand curve (Figure 14.2), representing the amount of people out of 350 who would be willing to use *SpoilerAlert!* as a function of cost in cents.

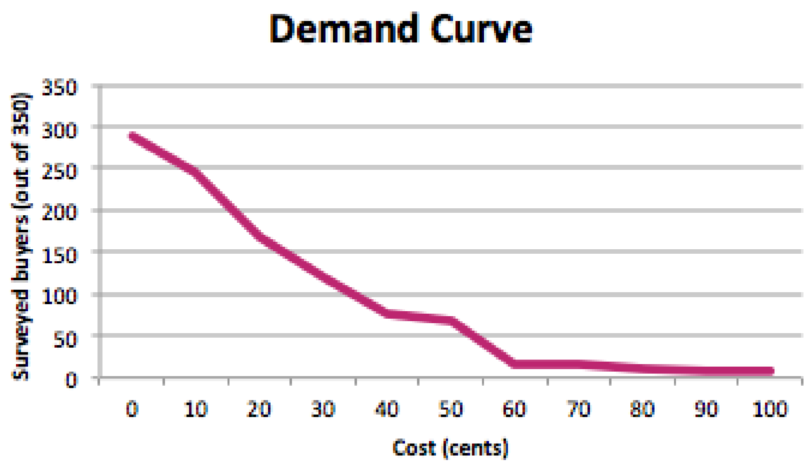


Figure 14.2. Demand curve of *SpoilerAlert!* by price, indicated in our market research survey.

Although *SpoilerAlert!* would get the most customers at the lowest price, profitability needs to take into account the price these customers are willing to pay to determine how much money SpoilSquad! would make from our product. Figure 14.3 shows the revenue from the projected sales based off the cost of *SpoilerAlert!*.

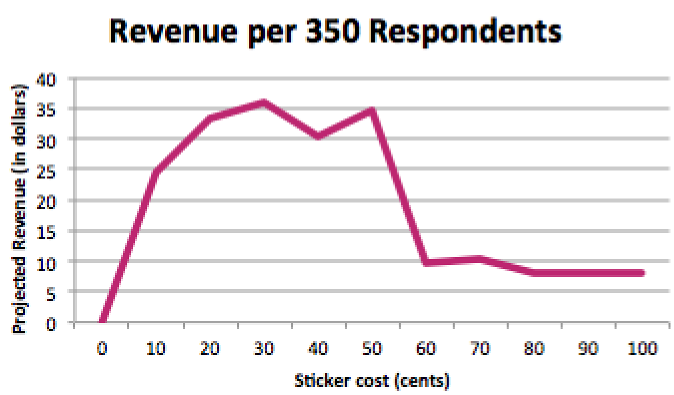


Figure 14.3. Revenue from *SpoilerAlert!* varying prices based on or market research survey.
varying prices based on or market research survey.

Based off the above graph, it seems the optimal price to charge is in between 20 cents and 30 cents. In addition, based off Washtenaw Dairy's Mary Jean Raab's expertise opinion of a 25-cent price, *SpoilerAlert!* has determined that 25 cents is the ideal price for *SpoilerAlert!*.

When subtracting the materials cost from the price, *SpoilerAlert!* yields a markup of 3788% and a profit of 24.3 cents. The University of Michigan's dairy producer and bottler, Prarie Farms, produces an estimate of 24 million jugs of milk. A partnership with Prarie Farms could yield an upside profit of $5,808,000. Any other milk suppliers that would be willing to implement *SpoilerAlert!* could yield very high profits.

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[4] "Paraffin (synthetic)", 21 C.F.R. §175.250 2018

[5] "Resinous and polymeric coatings", 21 C.F.R. §175.300 2018

[6] Center for Food Safety and Applied Nutrition. "Food Ingredients and Packaging Terms." *Food Ingredients and Packaging Terms,* U.S. Food and Drug Administration, 4 Jan. 2018