VALERIE TAY LIN WEI B. ENG. (HONS) CHEMICAL ENGINEERING JANUARY 2004

EFFECT OF SODIUM STEARATE (NAST) ON AIR FRESHENER TRANSPARENCY

VALERIE TAY LIN WEI

CHEMICAL ENGINEERING
UNIVERSITI TEKNOLOGI PETRONAS
JANUARY 2004

ABSTRACT

Sodium Stearate (NaSt) is produced in Stabilchem (M) Sdn. Bhd. via the fusion process between palm-based Fatty Acid (FA) and Sodium Hydroxide (NaOH). NaSt has a multitude of applications ranging from lubricating agents in wire drawing to emulsifying agents in the food industry. NaSt is also beginning to be used as a gelling agent in gel-based air fresheners. It is required that the NaSt used in the air freshener produce a translucent gel. In order to fulfill this industrial requirement, NaSt has to be tested for its performance in gel-based air fresheners. To do this, samples of NaSt with different chemical and physical properties are used to produce air fresheners. The air fresheners produced are then measured for transparency. The object of this project is to study the effect of chemical and physical properties of NaSt on gel air freshener transparency and to recommend the optimum NaSt parameters to be used in air fresheners. For this project, only the NaSt produced by Stabilchem (M) Sdn. Bhd. were used. To measure the gel transparency, a spectrophotometer called Hunter Lab Meter was used. Only three parameters were studied in this project, namely the effect of Free Fatty Acid (FFA) value and moisture content of the NaSt as well as the heating temperature when producing the air freshener. All the sample analysis laboratory test methods are that as stated in Stabilchem's Quality Control department. The air freshener formulation used was provided by a client of Stabilchem under confidentiality and trust. Therefore, the detailed formulation is not provided in this report. Among the components in the air freshener are D-Limonene, NaSt, Nonyl phenol and ethanol. After conducting this investigative project, it was found that gel air freshener transparency decreases slightly with increasing FFA values. The moisture content of NaSt has insignificant effect on gel transparency. Last but not least, gel transparency decreases as heating temperature is increased. From these findings, it can be concluded that Stabilchem's NaSt are capable of producing air freshener gels with satisfactory clarity as long as its chemical and physical properties are within the specifications set by the QC department. To ensure that the gel produced is always clear, the air freshener should be produced in a clean and dry area. Additives such as fragrance and colour can cause cloudiness. Therefore, compatible colouring and fragrance should be used in correct amounts to preserve the clarity of the air freshener gel.

ACKNOWLEDGEMENT

I would like to take this opportunity to thank several important people without whom this project would not have been a success.

First of all, my deepest gratitude to Mr. Lau Chee Chuan, Plant Manager of Stabilchem (M) Sdn. Bhd. for trusting me with this project and for his generosity in providing me with the necessary materials and advice to undertake this project.

Next, my thanks to Mr. Chia Kim Hooi, Stabilchem (M) Sdn. Bhd. Quality Control Chemist for allowing me to use the quality control laboratory for my experiments.

I would also like to thank Ms. Lim Chee Wee of Acidchem International for her guidance, advice and co-operation.

My deepest gratitude to Ms. Normawati M. Yunus for her expertise and time in guiding me through this project.

Last but not least, my thanks to the plant and laboratory technicians at Stabilchem (M) Sdn. Bhd. for their co-operation.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1
INTRODUCTION

Sodium Stearate (NaSt) is perhaps best known for its cleaning properties and is often used interchangeably with the term 'soap'. The earliest form of soap was made from animal fat (fatty acid) and did not contain any fragrance, nor does it produce any lather, which we take for granted in modern soaps. Today, natural soap is replaced with synthetic detergents. Not surprisingly the main component of these detergents is still NaSt. Thanks to advanced technology and the wonders of Research and Development, NaSt is no longer made exclusively from animal derived fatty acids. Equally effective and even more appealing to the general masses (especially vegetarians) are NaSt made from vegetable derived fatty acids. One of the pioneers in producing veggie-based fatty acids is Acidchem International Sdn, Bhd., which produces palm-based fatty acids and glycerin. These palm-based fatty acids are then purchased by Stabilchem (M) Sdn. Bhd. to produce metallic soaps or metal stearates.

1.1 PROJECT BACKGROUND

One of the many metal soaps produced by Stabilchem (M) Sdn. Bhd. is Sodium Stearate (NaSt). Due to the versatile and diverse characteristics of NaSt, its applications are not restricted to only soaps and detergents. NaSt now find applications in various industries as lubricating, emulsifying and gelling agents. Most of Stabilchem's NaSt clients are from the food, plastics and cosmetics industries.

This project focuses on NaSt used in gel air fresheners. Here, NaSt functions as a super clear gelling agent. In other words, NaSt is responsible for promoting the gelling of the components of the air freshener. It is desired that the NaSt used does not affect the colour or transparency of the final product.

In Stabilchem (M) Sdn. Bhd., NaSt is produced by directly reacting fatty acid with Sodium Hydroxide (NaOH) in an agitated vessel. A small amount of water is used to catalyse the reaction and promote heat release. The reaction between fatty acid and NaOH is a rather slow one. Often, the reactants need to be manually stirred to encourage heat release. Therefore, mixing may not be homogeneous at certain points in the reaction resulting in NaSt with varying properties. Due to this factor, the properties of the NaSt produced are often unpredictable.

To ensure that the NaSt sent for use in air fresheners perform satisfactorily, a performance test should be conducted. However, Stabilchem does not have a standard test for this. However, there is an analogous test for NaSt used in deodorant sticks. NaSt is also used as a gelling agent in deodorant sticks. 'Deodorant-grade' NaSt are tested by dissolving and heating the NaSt in ethylene glycol. The gel produced is then visually inspected for transparency. Since ethylene glycol is not one of the components in gel air fresheners, this test cannot be used for 'air freshener-grade' NaSt.

1.2 PROBLEM STATEMENT

1.2.1 Problem Identification

Recently, Stabilchem (M) Sdn. Bhd. began selling NaSt to be used in air fresheners. Two types of NaSt were produced, namely NaSt 200 and NaSt CP. These two differ in terms of physical appearance. NaSt 200 is a fine white powder while NaSt CP is in the form of coarse powder, which is lumpy and grainy to the touch.

Before Stabilchem can start selling the NaSt as a super clear gelling agent, the NaSt's performance in air fresheners must be ascertained first. When added into an air freshener formulation given by a customer, it was observed that NaSt within the same production run gave different degree of gel clarity. The reasons behind this must be determined in order to aid future production of NaSt to be used in air fresheners.

The possible causes of this problem can be divided into three categories namely the chemical properties of NaSt, the physical properties of NaSt and external factors. The chemical properties include the Free Fatty Acid (FFA), Free Alkalinity (FA) and moisture contents of the NaSt. The FFA content denotes the percentage of unreacted fatty acid in the NaSt. The FA content, on the other hand, represents the percentage of unreacted NaOH in the NaSt. Whether the NaSt has a FFA or FA vlue depends on the limiting reactant and the extent of reaction. As we can see, a sample of NaSt cannot have both FFA and FA values. The moisture content of NaSt is also known as Loss On Drying (LOD). This parameter tells us the percentage of water inside a sample of NaSt. The physical properties include the particle size of the NaSt sample. The only external factor taken into consideration in this project is the heating temperature used when producing the air freshener.

1.2.2 Significance of the Project

This project will give better understanding about the role of NaSt in air fresheners. In addition to that, this project intends to determine the various internal and external parameters that affect NaSt performance as a super clear gelling agent in air fresheners.

This project is important in determining the optimum NaSt parameters for air freshener production. In other words, this project will contribute to the determination of the most suitable grade of NaSt to be used in air fresheners.

1.3 PROJECT OBJECTIVES AND SCOPE

1.3.1 Objectives

The objectives of this project are:

i. To determine the factors that affect NaSt transparency in air fresheners.
ii. To recommend the optimum NaSt parameters for producing air fresheners.

1.3.2 Project Scope

The main interest of this project lies in the effect of Free Fatty Acid (FFA) value, moisture content (LOD) and heating temperature on gel-based air fresheners' transparency. The transparency is measured using the Hunter Lab Meter DP-9000 Version VI.32.

The NaSt used in this project is solely supplied by Stabilchem (M) Sdn. Bhd.

The colour of the air freshener and whether it remains gelled after a period of time will not be of concern in this project.

Other external factors such as climate, humidity, plant contamination and raw material quality will not be studied in this project.

CHAPTER 2
LITERATURE REVIEW

2.1 AIR FRESHENERS

2.1.1 History of Air Fresheners

An air freshener is a product designed to mask unpleasant odours by delivering fragrance and other odour counteractants into the air. Fragrance compounds have been used to freshen air and mask odours since ancient times.

Over the last 2,000 years a variety of spices and floral extracts have been used to impart pleasant aroma. However, it was not until 1948 that the first modern air freshener was introduced. This product was a pressurized spray containing about 1% perfume, 24% alcohol or other solvents, and 75% chlorofluorocarbon (CFC) propellant. This was able to deliver a fine mist of fragrance that remained suspended in the air for a long period of time.

However, due to increasing concern over the effect of CFCs on the ozone, new forms of air fresheners were produced. The most popular and innovative format was the "plug-in", whose source of fragrance is a gel like fragrance concentrate. This gel air freshener is now a common sight in homes as well as automobiles.

One of the key components in this gel air freshener is Sodium Stearate (NaSt). As mentioned above, NaSt is used to promote the gelling of the air freshener.

2.1.2 Components of Air Freshener

A simple gel air freshener comprises of three basic components, namely fragrance, solvent and gelling agent. Commercial air fresheners usually contain other chemicals such as colouring and additives to improve texture, transparency and life span of the air freshener.

In this project, the air freshener will be produced from D-Limonene, Nonyl Phenol 9, Ethanol, NaSt and de-ionised water (DI water).

D-Limonene

D-Limonene (commercially known as citronella) is the major component of the oil extracted from citrus rind. When citrus fruits are juiced, the oil is pressed out of the rind. This oil is separated from the juice, and distilled to recover certain flavor and fragrance compounds. The bulk of the oil is left behind and collected. This is food grade d-limonene.

After the juicing process, the peels are conveyed to a steam extractor. This extracts more of the oil from the peel. When the steam is condensed, a layer of oil floats on the surface of the condensed water. This is technical grade D-limonene. D-Limonene can be used either as a straight solvent, or as a water dilutable product[1].

D-Limonene has a distinct orange odour and an oily, viscous texture. In this project, D-Limonene is used as the source of fragrance. Below is the molecular structure of D-Limonene or 4-isopropenyl-1-methylcyclohexnene.

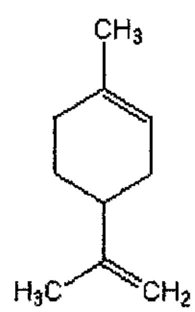


Figure 1: Molecular structure of D-Limonene

Nonyl Phenol

Nonyl Phenol has a molecular formula of $C_{15}H_{24}O$. It comprises of a mixture of monoalkyl phenols and exists as thick, light yellow, straw color liquid with slight characteristic phenolic odor. It is poorly soluble in water but soluble in alcohols[2].

Nonyl Phenol is used in a variety of processes and products. Its many uses include lubricating oil and grease additives, agricultural chemicals, resin and rubber additives, light stabilizers, plasticizers, wetting and antifoam agents, epoxy resin diluents, and surfactants.

In this project, Nonyl Phenol is used as a clarifying agent. Below is the molecular structure of Nonyl Phenol:

Figure 2: Molecular structure of Nonyl Phenol

Ethanol

Ethanol, CH_3CH_2OH, is an alcohol, a group of chemical compounds whose molecules contain a hydroxyl group, –OH, bonded to a carbon atom. Ethanol or ethyl alcohol is a clear, colorless liquid with a characteristic, agreeable odor. In dilute aqueous solution, it has a somewhat sweet flavor, but in more concentrated solutions it has a burning taste[3].

Ethanol has high polarity and is widely used as a solvent for salt-like compounds. Ethanol is also vapourises readily due to its low vapour pressure and boiling point. In this project, Ethanol functions as a solvent and fragrance-releasing agent when it vapourises. Below is the molecular structure of Ethanol:

$$\begin{matrix} & H & & & & \\ H_3C - & C & - & O & - & H \\ & H & & & & \end{matrix}$$

Figure 3: Molecular structure of Ethanol

Sodium Stearate

NaSt is an organometallic compound with a molecular structure of $C_{18}H_{35}NaO_2$. NaSt can be said to consist of two parts, namely the hydrophobic carbon chain and the hydrophilic sodium cation[4].

NaSt exists in the form of white powder with a soapy feel. It is sparingly soluble in cold water and polar solvents but dissolves freely in hot solvents. NaSt solutions in hot solvents form gels upon cooling[5].

Among the common usage of NaSt were mentioned in the Introduction section.

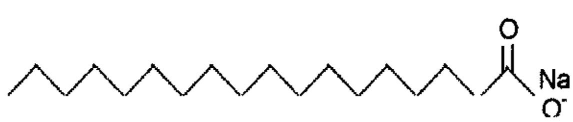


Figure 4: Molecular structure of NaSt

2.2 PRODUCTION OF SODIUM STEARATE

As mentioned in the Introduction, Sodium Stearate (NaSt) is produced by directly reacting fatty acid (stearic acid) and Sodium Hydroxide (NaOH). The reaction is governed by the chemical equation given below:

$$\underset{\text{Fatty acid}}{RCOOH} + \underset{\text{Sodium Hydroxide}}{NaOH} \longrightarrow \underset{\text{Sodium Stearate}}{Na(RCOO)} + \underset{\text{Water}}{H_2O}$$

In Stabilchem (M) Sdn. Bhd., NaSt is produced via a method called the dry/fusion process. Palm-based fatty acid flakes (with an average carbon chain length of C-18) is reacted with NaOH 'pearls' (with average purity of 98%) in a jacketed agitated vessel. The vessel can be heated up or cooled down by means of steam and cooling water supplies to the jacket.

The fatty acid flakes and NaOH are heated and mixed in the vessel at temperatures ranging from 70°C - 120°C. Once melted, the fatty acid-NaOH mixture will exist in the form of white liquid mixture. Upon the addition of water, a certain degree of heat release in the form of steam will be observed. The liquid mixture will gradually become drier, first forming lumps in the mixture, followed by a dough-like mixture and eventually white powder. This reaction is somewhat time consuming and will only form powder with extensive agitation and cooling. In this dry/fusion process, water serves as a catalyst as well as a heat-releasing agent.

As mentioned earlier in Chapter 1: Introduction, manual stirring is often needed to aid heat release. This happens when the reactants are in the dough-like state. The 'dough' formed is somewhat lumpy and will tend to stick together in a big lump around the agitator. This makes the mixing provided by the agitator inadequate, therefore hindering heat release, which is crucial for the reactants to turn into NaSt powder. This is where the manual stirring come in. Although this additional stirring helps heat release to a certain extent, stirring by hand is, needless to say, non-uniform. As a result, the mixing between reactants at this point may not be homogeneous leading to incomplete reaction at certain points in the vessel. This explains why the NaSt produced is sometimes acidic (having FFA value) and sometimes alkaline (having FA value).

From the vessel, a small amount of sample is sent to the Quality Control laboratory for testing. Vessel samples are typically tested for Free Fatty Acid/ Free Alkalinity and moisture contents.

Once approved by the QC, the product from the vessel is sent to a cooler where the powder is cooled to roughly 60 °C. Rotating blades in the cooler breaks the powdery chunks into smaller pieces before being sent for milling.

At the mill, the NaSt chunks are grinded to the desired fineness. The fineness of grind can be controlled via three parameters, namely the feed rate of the NaSt to the mill, the classifier speed and the inlet/ outlet air valve opening. The fine powder is then sent to a bag filter/ bag house where the powder is separated from the carrier air. The powder is purged from the bag house using compressed air at 6 bar. The NaSt powder is now considered as the finished product and is stored in a product silo.

Random samples of the finished NaSt are collected and sent for QC testing. Approved samples are ready to be shipped while rejected samples are either reprocessed or sold as lower grade products. Refer to Appendix I for the process flow chart of NaSt production.

2.3 GELLING AGENTS

As the name suggests, gelling agents are chemicals that promote the gelling of other materials. Gelling agents are usually large molecules that dissolve in hot water or solvents.

Gelling agents function by forming structures/ network that retain water/ solvent in the spaces between its molecules. Gelling agents are also known as viscosity modifiers or thickeners.

2.3.1 Gels

A gel is defined as a colloid in which the dispersed phase has combined with the continuous phase to produce a viscous jelly-like product. In gels, the dispersed phase is liquid while the continuous phase is solid.

A gel is made by cooling a solution whereupon certain kinds of solids form submicroscopic crystalline particle groups that retain much of the solvent in the interstices[6]. In other words, gels are liquid-water-containing networks showing solid-like behavior with characteristic strength[7].

Gels are usually transparent but may become opalescent. Most gels exhibit a property called thixotropy. A thixotropic gel appears to be solid and maintains a shape of its own until it is subjected to a shearing (lateral) force or some other disturbance, such as shaking. It then acts as a sol (a semi fluid colloid) and flows freely. Thixotropic behavior is reversible, and when allowed to stand undisturbed the sol slowly reverts to a gel.

2.3.2 Examples of Gelling Agents

Hydrocolloids

A group of chemical compounds that may be of particular interest in this project are hydrocolloids. Hydrocolloids are hydrophilic polymers, of vegetable, animal, microbial or synthetic origin, that generally contain many hydroxyl groups and may be polyelectrolytes. They are naturally present or added to control the functional properties of aqueous materials. Most important amongst these properties are viscosity (including thickening and gelling) and water binding. Other properties that are controlled by hydrocolloids include emulsion stabilization, prevention of ice recrystallization and organoleptic properties.

All hydrocolloids interact with water, reducing its diffusion and stabilizing its presence. This interaction depends on the formation of hydrogen bonding. As hydrocolloids can dramatically affect the flow behavior of many times their own weight of water, most hydrocolloids are used to increase viscosity, which is used to stabilize foodstuffs by preventing settling, phase separation, foam collapse and crystallization. Viscosity generally changes with concentration, temperature and shear strain rate in a complex manner dependent on the hydrocolloid and other materials present. Mixtures of hydrocolloids may act synergically to increase viscosity or antagonistically to reduce it.

Many hydrocolloids form gel. The characteristic strength of the gel formed is influenced by hardness and brittleness, which in turn are dependent on the structure of the hydrocolloid present. Hydrocolloids gel when intra- or inter-molecular hydrogen bonding is favored over hydrogen bonding to water to a sufficient extent to overcome the entropy cost. Often the hydrocolloids exhibit a delicate balance between hydrophobicity and hydrophilicity. Extended hydrocolloids tend to tangle at high concentrations and its molecules will wrap around each other, forming helical junction zones, which trap water in the interstices[7].

Two of the most commonly found hydrocolloids are agar and gelatin. The gelling behaviour of these two compounds will be discussed below.

Agar

Agar is prepared from red seaweeds and consists mainly of agarose and agaropectin. The former is a linear polymer while the latter is a mixture of smaller molecules. The gelling abilities of agar is dominated by agarose, which is an excellent gelling agent compared to agaropectin.

Agar is insoluble in cold water but dissolves readily in hot water. It forms random coils in the hot solution, which will tangle and form the helical gel network upon cooling.

The gel network of agarose contains double helices formed from left-handed threefold helices. These double helices are stabilized by the presence of water molecules bound inside the double helical cavity. Exterior hydroxyl groups allow aggregation of up to 10,000 of these helices to form suprafibers[7]. The gel structure of agar is shown in Figure 5 below:

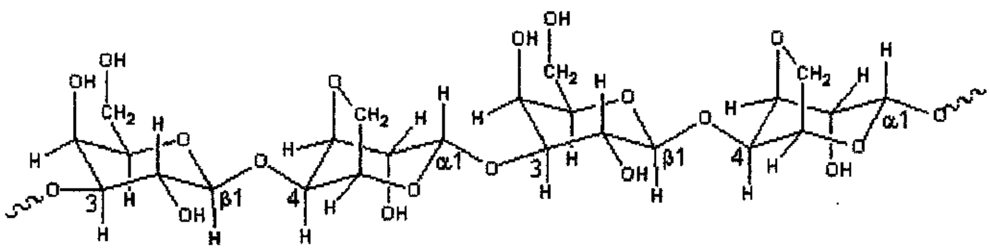


Figure 5: Molecular structure of agar gel network

Gelatin

Gelatin is prepared from collagen extracted form the skin and bones of animals. Gelatin contains a large number of glycine, proline and 4-hydroxyproline residues. A typical structure is -Ala-Gly-Pro-Arg-Gly-Glu-4Hyp-Gly-Pro-.

Gelatin consists of extended left-handed proline helix conformation in single or multi-stranded polypeptides. Solutions undergo coil-helix transition followed by aggregation of the helices[7].

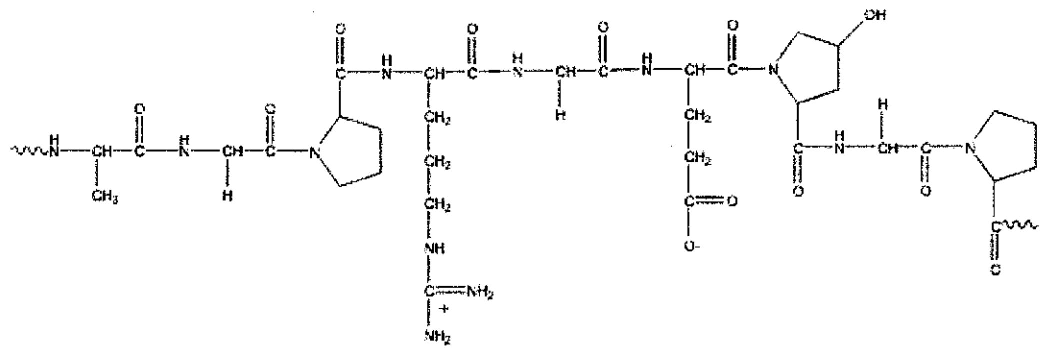


Figure 6: Molecular structure of gelatin gel network

2.3.3 Sodium Stearate as a Gelling Agent

NaSt can be likened to hydrocolloids in the sense that NaSt is a large molecule of vegetable. Although NaSt is not a hydrophilic polymer, it is a polar molecule that contains both hydrophobic and hydrophilic counterparts. Hydrocolloids contain a large number of hydroxyl (OH) groups, a trait that gives hydrocolloids its hydrophilicity. It is the presence of these hydroxyls that allow hydrogen bonding with water molecules that is so characteristic of hydrocolloids. In NaSt, it is suspected that instead of hydroxyls, the functional group that encourages interaction with water and other solvents is the negatively charged sodium. This cation is also the hydrophilic part of the NaSt molecule. In detergents and soaps, it serves to lower the surface tension of water.

It has been observed that NaSt will tend to gel after being dissolved in water and ethanol. This is not surprising as NaSt is a polar molecule and will therefore interact readily with other polar substances.

In the air freshener formulation used, NaSt, Nonyl phenol and Ethanol are polar substances. Therefore, in the similar manner that hydrocolloids form gels, it can be said that the NaSt dissolves in these solvents at high temperature to form long molecules that tangle with each other. These tangled NaSt molecules may form helixes similar to that formed by hydrocolloids that trap the solvents and D-Limonene in its interstices.

2.4 HUNTER LAB METER (SPECTROPHOTOMETER)

As mentioned earlier, the object of this project is to measure the transparency of the air freshener gel formed by NaSt. The analytical instrument that will be used to measure transparency is the Hunter Lab Meter (refer to Appendix III for pictures of Hunterlab Meters). This meter functions on the same principle as spectrophotometers.

2.4.1 Theory

The spectrophotometer is built based on the theory that each material has its own characteristic absorbance. This means that different materials will have different capabilities of absorbing lights with varying wavelengths. The amount of light absorbed depends on the type of sample. How much or how little light passes through the sample is a measure of the transparency or opacity of the sample material.

2.4.2 Operating Principle

A spectrophotometer consists of a light source, a sample cell, a reference cell, a photo detector and a computer to analyse the light transmittance.

The analysis begins from the light source. Different types of lamps are available to deliver lights with varying wavelengths. The analytical wavelength is chosen based on the absorbance characteristics of the analyte. The light source also contains a monochromator, which receives polychromatic lights (lights with multiple wavelengths) from the source lamp and allows only monochromatic lights to exit.

The Hunter Lab Meter is capable of analysing sample opacity, colour and concentration. For this project, the meter is set to analyse the opacity of the sample, which is given in percentage of light that does not pass through the sample. Transparency of the sample is then 100% minus the opacity reading.

To analyse an analyte, the sample is placed on the light path. The amount of light that passes through the sample is sent to the photo detector to be analysed[8]. The opacity of the sample is calculated internally and then displayed on the display panel of the meter. For the detailed method of using the Hunter Lab Meter, please refer to the experimental methods in Chapter 3.

As with any analytical instruments, the Hunter Lab Meter's accuracy may be compromised due to errors. Errors in measurements can be divided into two categories, namely instrument related and non-instrumental related.

A major source of instrument related errors is stray light. Stray light is the unwanted component of radiant energy outside the spectral bandwidth. The main source of stray light is the dispersing element in the monochromator. Other sources of stray light include scattering of light and unwanted reflections from other optical elements as well as light leaks in the instrument. A well-constructed instrument will have negligible light leaks. Another instrument related error is electronic noise in the detector. This 'noise' causes random fluctuations of the photon beam reaching the detector.

Non-instrumental errors usually originate from the nature of the analyte. Several analyte-related factors include reflection of air/cell and analyte/cell interfaces, scattering by suspended particles and absorption of light beam by the analyte. Additional fluorescence component (re-emitted absorbed energy) can also contribute to non-instrumental error.

CHAPTER 3

METHODOLOGY

3.1 EXPERIMENTAL PROCEDURES

3.1.1 Air Freshener production

Apparatus:

1) 200ml beaker.
2) Hot plate.
3) Stirrer.
4) Thermometer.
5) Petri dish.
6) Shrink wrap.

Reagents:
1) NaSt.
2) D-Limonene.
3) Nonyl Phenol 9.
4) Ethanol 95%.
5) Distilled water.

Procedure:

First, the D-Limonene is heated to about 70-75°C using a hot plate. Sodium Stearate is then added in. The mixture stirred and heated until all the Sodium Stearate has dissolved. Next, Nonyl Phenol 9, Ethanol 95% and distilled water are added in. The stirring and heating are to be stopped as soon as the solution turns clear. Finally, the mixture is poured into a petri dish and cover with shrink wrap. The mixture is left to cool.

**Note: The detailed formulation is withheld due to company trade secret.*

3.1.2 Transparency Test

Apparatus:

1) Hunter Lab Meter.

Procedure:

First, the Hunter Lab Meter is switched on and left to warm up for 30 minutes. After warming up, the mode of the meter is set to 'Opacity'. To calibrate, a black tile is first placed on the light path and 'Read' button is activated. When prompted, the black tile is removed and replaced by a white tile. The 'Read' button is pressed again. Next, a gel sample is placed on the light path and the top of the sample is covered with the black tile. Press 'Read'. After that, the white tile is placed on top of the sample and press 'Read'. The opacity of the sample will appear on the display screen.

3.2 TOOLS AND CHEMICALS USED

3.2.1 Apparatus/ Equipment

1) 200ml beaker.
2) Round bottom flack.
3) Thermometer.
4) Stirrer.
5) Hot plate.
6) Analytical balance.
7) Manual sieve.
8) Infra-red moisture balance.

3.2.2 Reagents/ Samples

1) NaSt 200.
2) NaSt CP.
3) D-Limonene.
4) Nonyl Phenol 9.
5) Ethanol 95%.
6) Phenolphthalein.
7) 0.1M NaOH.
8) Fatty Acid 70-18.

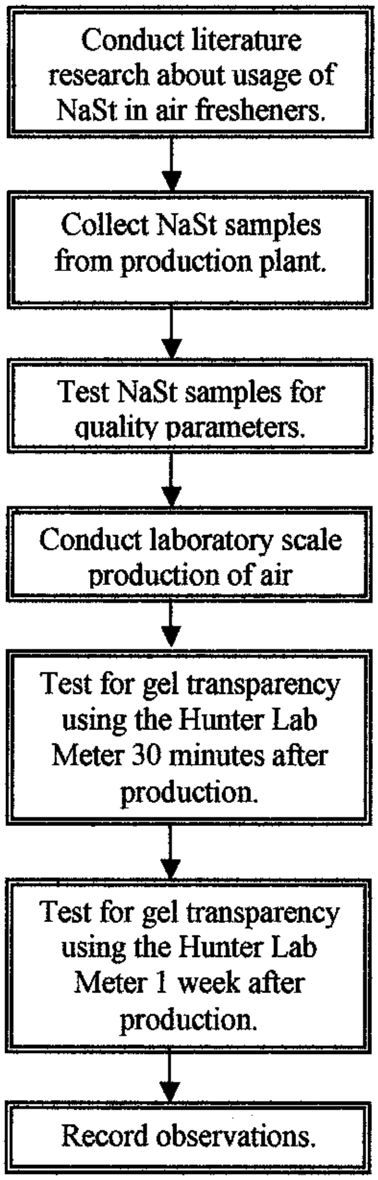


Figure 7: Flow chart for overall project methodology

CHAPTER 4

RESULTS AND DISCUSSION

4.1 OBSERVATIONS

Some observations were made when conducting the laboratory scale production of gel air freshener:

i. D-Limonene heats up readily, reaching a temperature of 60-80°C within 15 minutes.

ii. NaSt 200 does not disperse readily in D-Limonene. Upon addition of NaSt, the D-Limonene turns cloudy and the fine NaSt powder tends to form small lumps.

iii. The agitation provided by the stirrer is insufficient to break up the NaSt lumps. This has to be done manually.

iv. NaSt CP disperses readily in D-Limonene and does not form lumps.

v. The solution turns slightly clearer upon the addition of Nonyl Phenol.

vi. Solution turns a clear pale yellow upon the addition of Ethanol and De-ionised (DI) water.

vii. Even after the solution has turned clear, lumps of NaSt remains in the solution (this is observed for NaSt 200).

viii. Once the stirring is stopped, a clear skin-like layer immediately forms on the surface of the solution.

ix. Upon cooling, the solution gels. The surface of the gel has an oily feel.

x. The gel air freshener produced using the NaSt with LOD=0% is softer and less firm compared to the rest.

xi. At temperatures between 90°C-110°C, the D-Limonene-NaSt mixture is a cloudy yellow colour and has a sticky texture.

xii. Gel solution takes longer to gel at higher temperatures.

xiii. Gel solutions prepared at high temperatures are more dilute and have less bubbles.

4.2 FREE FATTY ACID (FFA) AND MOISTURE CONTENTS (LOD) OF NAST

As mentioned earlier, NaSt is used as a gelling agent in gel-based air fresheners. Stabilchem (M) Sdn. Bhd. has only recently started producing NaSt to cater for this application. In order to ensure that the NaSt produced perform satisfactorily, the transparency of the gel produced must be tested. Customers always require that the gel produced by any gelling agent is initially clear. Any colour for aesthetical purposes are only later added.

In the Problem Statement it was also mentioned that the NaSt produced via the fusion process in Stabilchem tend to have varying chemical and physical properties. These variations may affect the clarity of the gel produced.

To go about investigating the effects of NaSt properties on its performance, samples must first be collected from the production plant. During the NaSt campaign run from August to September 2003, NaSt 200 and NaSt CP were produced for customers in various industries. These two grades of NaSt differ in terms of particle sizes. NaSt 200 is grinded to a fineness of 200 mesh (sieve size of 200 perforations per square inch) while NaSt CP is grinded to 100 mesh (ungrounded NaSt are also produced in Stabilchem). The fineness of the grind are measured using an analytical equipment called Mastersizer and the results are termed 'Granulometry' or GN for short. The other parameters such as Free Fatty Acid (FFA) value, moisture content, ash content and bulk density are within similar ranges.

The samples were collected with the help of Stabilchem's Quality Control Department. 6 pellets of NaSt 200 (Lots ADC 0057 and 0058) with varying FFA values and moisture contents were collected. These pellets had FFA values ranging from 0.4%-1.38% while their moisture contents ranged from 0.9%-1.4%. The GN of these 6 samples are close.

2 pellets of NaSt CP (Lot ADC0055) were collected. These two samples were collected primarily to compare the performance of NaSt with different particle sizes in air fresheners.

Before beginning the laboratory scale production of air fresheners, all these samples were analysed again for their chemical and physical properties. The experimental data for sample analysis can be found in Appendix IV. It was found that the results obtained by the student tallies with that recorded in the QC laboratory.

Table 1: Free Fatty Acid (FFA) and Moisture content (LOD) for NaSt 200.

Lot number	Pellet number	FFA %	LOD %	GN %
ADC 0057	#7	1.00	1.4	97.9
ADC 0075	#21	0.48	0.9	96.5
ADC 0058	#10	0.96	1.3	96.8
ADC 0058	#12	0.55	1.4	96.5
ADC 0058	#18	0.4	1.0	96.8
ADC 0058	#19	1.38	1.0	97.1
ADC 0055	#12	1.13	1.3	96.18
ADC 0055	#14	0.85	1.0	94.22

4.2 TRANSPARENCY TEST

The first round of air freshener production and testing was done on the NaSt 200 and NaSt CP samples mentioned above. This round is to establish a baseline for the NaSt performance. The production of air freshener requires continuous heating and stirring. As mentioned in Observations, the NaSt tends to lump together in the D-Limonene solution and does not disperse as desired. Even in its dry powder form, the NaSt form lumps that can be broken by touch. The formation of these lumps is due to the fact the NaSt 200 powder is very fine and tends to pack together rather effectively when left in a bag. Since D-Limonene is a rather viscous liquid, it can be said that the lumps in the NaSt sample cannot break and disperse even though agitation in provided by the stirrer. To ensure a homogeneous air freshener solution, the lumps have to be broken manually using a spatula. Not surprisingly, NaSt CP does not have this problem probably because its particles are coarser and do not aggregate as much. Upon addition of Nonyl Phenol, Ethanol and DI water, the solution turn clear. This is when gelation takes place. Gelation is the process where the gelling agent absorbs solvent, swells and forms a 3D helical structure. The onset of gelation is characterized by an increase in solution viscosity and clarity[9]. Therefore, it can be said that NaSt (gelling agent) absorbs the DI water and possibly Nonyl Phenol and Ethanol to increase the viscosity of the air freshener solution, thus forming a gel. It was also observed that the gel has an oily feel. This is because the fragrance and chemicals used are organic compounds and present in ambient temperatures as viscous, oily liquids.

After the air fresheners have cooled and gelled, the gels were measured for transparency using the Hunter Lab Meter. This spectrophotometer gives the reading in terms of opacity, which is the percentage of source light that do not pass through the sample. Because the sample surface may not be even enough to provide an accurate reading, measurements were taken at four different angles for a single gel sample.

Table 2: Transparencies for NaSt 200 and NaSt CP.

Lot number	Pellet number	FFA %	LOD %	GN %	Transparency (%)	
					Fresh sample	One-week-old sample
ADC0057	7	1.0	1.4	97.9	93.33	61.72
ADC0057	21	0.48	0.9	96.5	91.11	57.29
ADC0058	10	0.96	1.3	96.8	90.45	64.00
ADC0058	12	0.55	1.4	96.5	90.52	65.14
ADC0058	18	0.4	1.0	96.8	95.60	72.35
ADC0058	19	1.38	1.0	97.1	92.86	55.35
ADC0055	12	1.13	1.3	96.18	87.38	86.63
ADC0055	14	0.85	1.0	94.22	92.13	87.47

From the results obtained (refer to Table 2), we see that the transparencies of the 8 samples produced vary quite markedly. In an attempt to find a relationship between FFA, LOD (moisture content) and GN, the transparencies were plotted against each parameter (refer to Figures 8, 9 and 10).

From Figure 8, it can be seen that as the FFA increases up to 0.96%, there is a slight decrease in gel transparency. This observation is not surprising since fatty acids

(FA) are white coloured solids in room temperature. Therefore, if a sample of NaSt has excess FA, the FA will show up when the gel cools, thus causing the gel to be cloudy. However, as the FFA value continues to increase, the transparency of the gel seems to improve. This inconsistency could be due to other parameters of the sample. When the measurements were taken again one week later, it was observed that the gel samples have deteriorated badly. This could be due to insufficient sealing and improper storage of the samples. Gels in general have been found to be vulnerable to dehydrating environments. Loss of moisture from the gel matrix causes the gel to shrink and lose its clarity. Nevertheless, looking at the measurements taken, it can be seen that gel transparency generally decreases with increasing FFA value. From this observation, it can be speculated that FFA values of NaSt affects gel clarity.

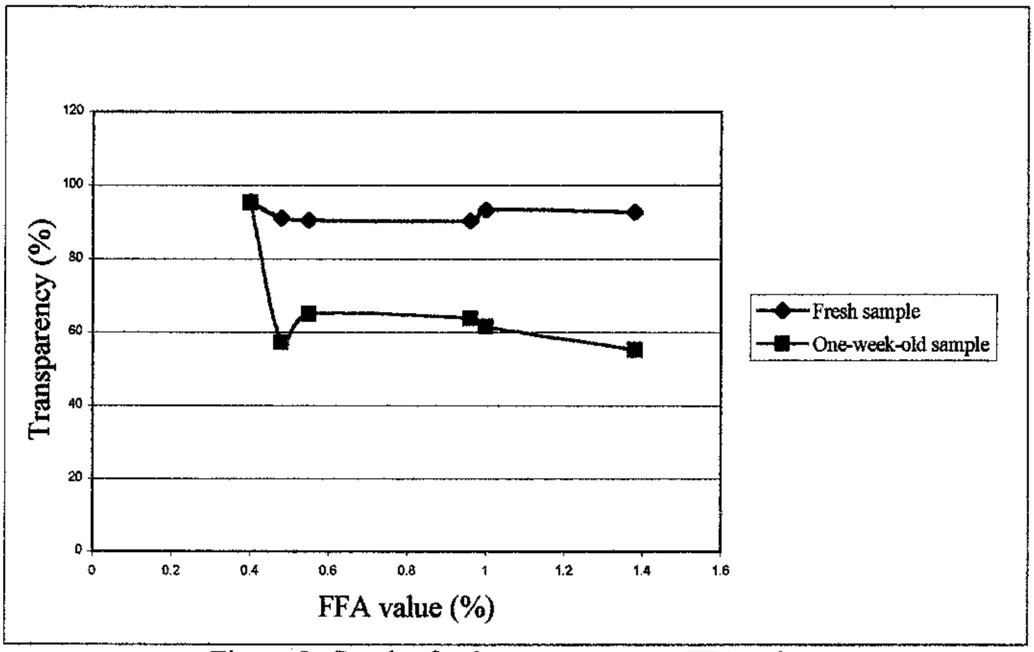


Figure 8: Graph of gel transparency vs FFA value.

From Figure 9, no obvious trend or relationship can be seen between gel transparency and the moisture content of NaSt. However, it has been stated in literature that the essence of the gelling mechanism is the absorption of water/ solvent by the gelling agent[9]. Therefore, the lack of water in a sample should have an effect on the gelling capability of the NaSt. Again, the fluctuating measurements could be due to other interfering parameters or instrument error as mentioned in section 2.4.2.

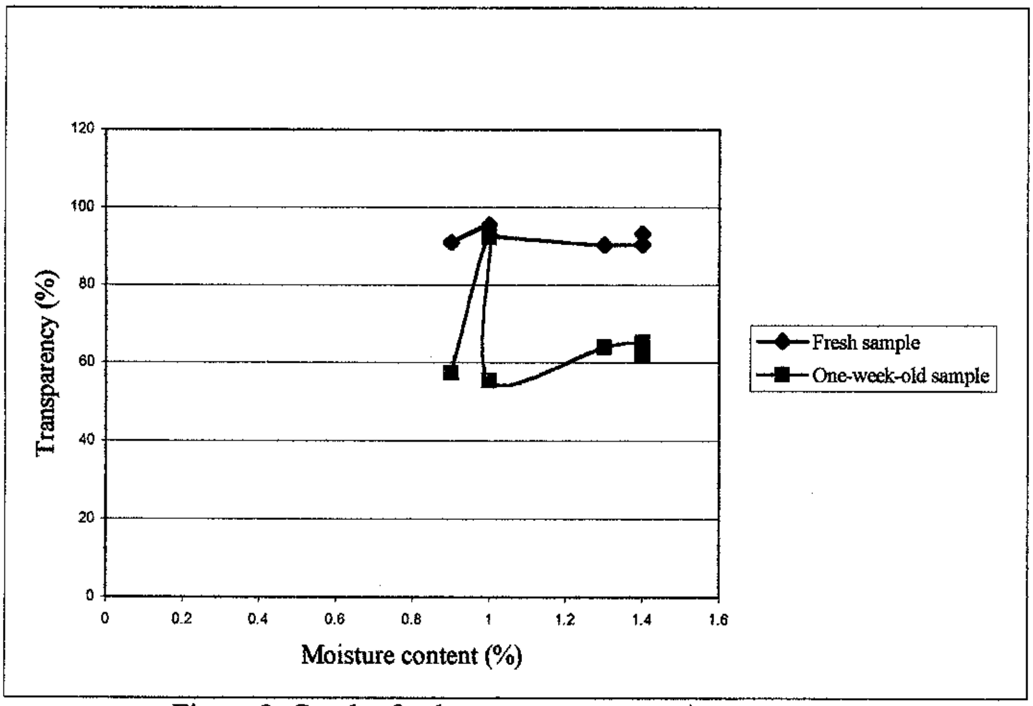


Figure 9: Graph of gel transparency vs moisture content.

Figure 10 attempts to draw a relationship between gel transparency and particle size. Common sense dictates that the finer the particles, the easier it is for the powder to disperse, therefore the better the gel clarity. In addition, bigger particles are coarser and tend to provide more resistance to light traveling though the gel sample, therefore giving the gel poorer clarity. However, looking at the curve obtained in Figure 10, we see no apparent relationship between transparency and particle size. This could be due to the fact that the particle size range available is very narrow. In order to see a more substantial relationship between clarity and particle size, a larger range is needed. The Research and Development Department of Acidchem International Sdn. Bhd. has done a similar test comparing NaSt 200 and ungrounded NaSt CP. This range is definitely larger than that available in Figure 10 and is more suitable for comparison. It was reported that the gel produced using the ungrounded NaSt CP was cloudy and opaque. This confirms the above reasoning that gel transparency decreases with increasing particle sizes.

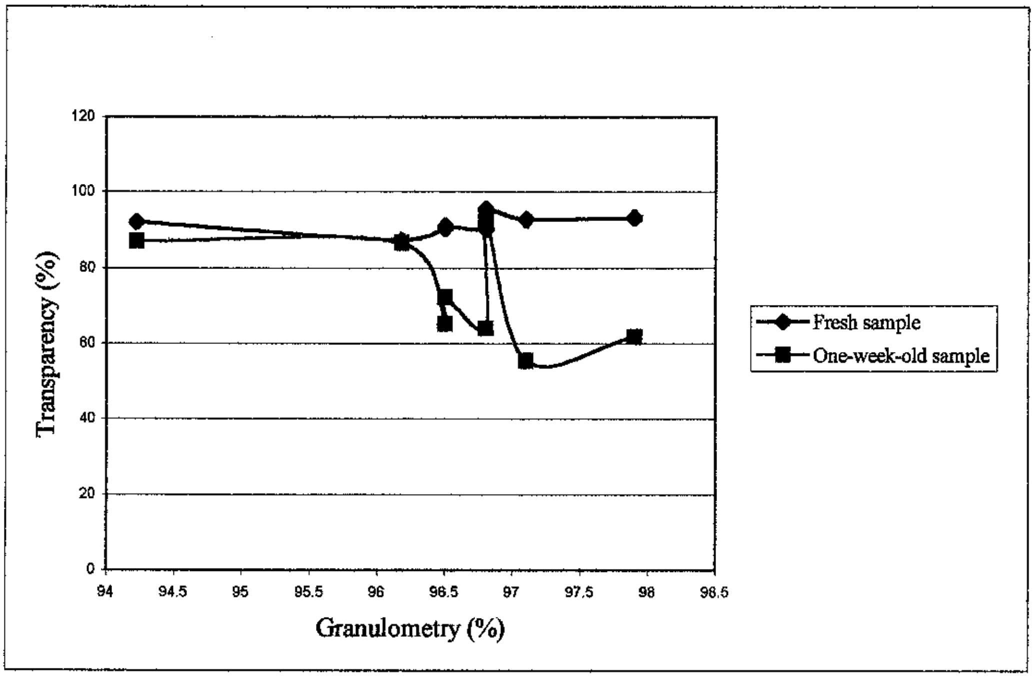


Figure 10: Graph of gel transparency vs particle size.

4.2.1 Effect of FFA Values on Gel Transparency

As mentioned earlier, this first round of tests was done to establish a baseline for NaSt performance. From Table 2, we see that NaSt 200 from pellet #18 of lot ADC0058 has the best transparency among the 8 samples collected. Therefore, this pellet is used for the consequent studies.

From Figure 8, it was observed that the FFA values of NaSt have an effect on gel transparency. To confirm this, the NaSt from pellet #18 was spiked with increasing amounts of fatty acid (FA). To avoid the differences in size between the NaSt and FA to interfere with the results, the FA was grinded and sieved to 200 mesh. This fine FA powders were then added into the NaSt in 1%, 3% and 5% excess. From Table 3 and Figure 11, it can be observed that gel transparency will generally decrease with increasing FFA values. The measurements taken one week later also exhibit the same trend. It must be noted, however, that the difference in clarity as the FFA values increases is very slight (in the order of 2%). In other words, the FFA value of NaSt can increase up to 5% and still provide acceptable gel clarity when used in air fresheners. But since the maximum allowable FFA value for NaSt as specified by QC is 2%, the effect of FFA on gel clarity should not be of major concern.

Table 3: Transparencies of air fresheners produced using NaSt with excess FA.

Sample description	Weight of NaSt (g)	Weight of FA (g)	Transparency	
			Fresh sample	One-week-old sample
1% excess FA	4.9505	0.0504	96.35	90.95
3% excess FA	4.8500	0.1500	97.18	92.38
5% excess FA	4.7504	0.2503	95.13	90.17

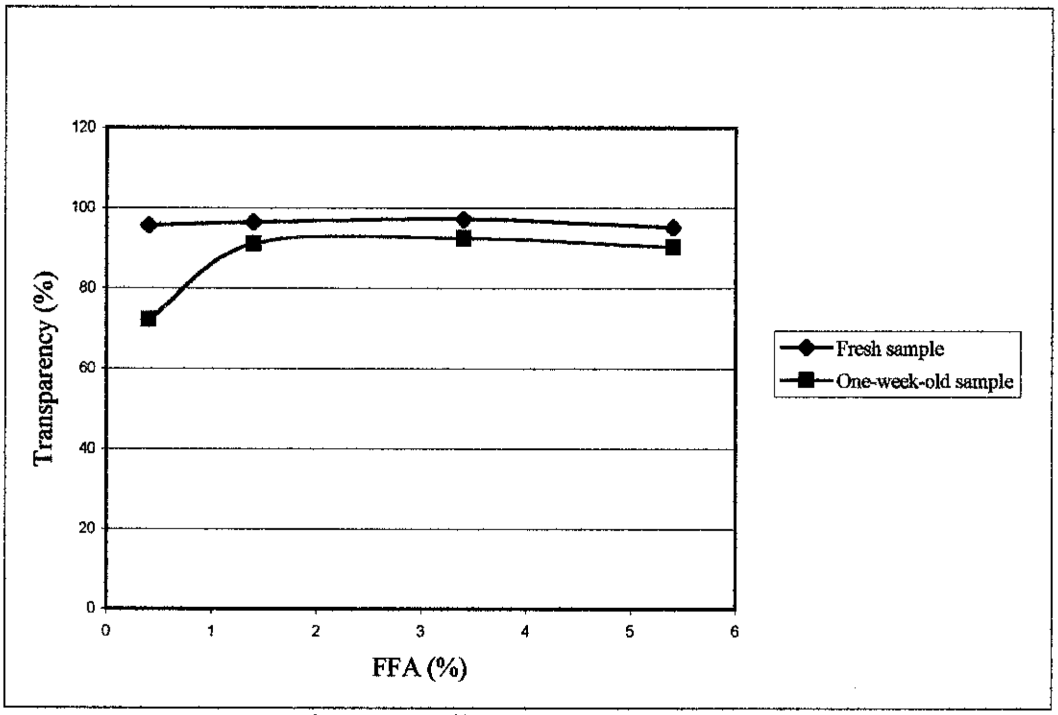


Figure 11: Effect of FFA on gel transparency.

4.2.2 Effect of Moisture Content on Gel Transparency

To study the effect of moisture content on air freshener gel transparency, samples of NaSt from pellet #18 were oven dried for different periods of time to produce samples with varying moisture contents. From Table 4 and Figure 12, it was observed that gel clarity decreases with decreasing moisture contents. As mentioned earlier, the onset of gelation is when there is an increase in solution clarity and viscosity. This happens when the gelling agent absorbs water/ solvent and forms a helical network to trap the moisture/ solvent. Gel solution becomes clear upon gelling because the water is transferred from the liquid solution into the solid gel network[9]. Therefore, when a sample has low moisture content, there would be less water to absorb. As a result, the gelling agent does not swell to its full capacity. And since there is less water to be absorbed from the liquid phase into the gel network, the increase in clarity will be less. It must be noted that the presence of moisture causes cloudiness in gels[11]. This statement is of course the opposite of the observation made here. But the moisture mentioned in Ref. 11 refers to external moisture, which exists as an impurity during the gelation process. This means any moisture on the petri dish will cause cloudiness. It was observed that the gel produced using the NaSt sample with 0% moisture is less firm compared to the rest. This phenomena can also be explained using the fact that and increase in solution viscosity is an important benchmark in the gelling process. Since the NaSt has virtually no moisture, it can be said that the gelling agent cannot swell and thicken the solution. Therefore, the gel produced is slightly less viscous than normal. From the above results and observations, it can be concluded that the moisture content of the NaSt not only have an effect on gel transparency but also its strength and viscosity. However, the decrease in gel clarity is in the order of 10^{-2} and can be considered as insignificant.

Table 4: Transparencies of air freshener produced using NaSt with varying LOD.

LOD %	Transparency	
	Fresh sample	One-week-old sample
1.0	95.60	72.35
0.8	95.54	90.24
0.3	95.42	86.52
0	95.21	80.70

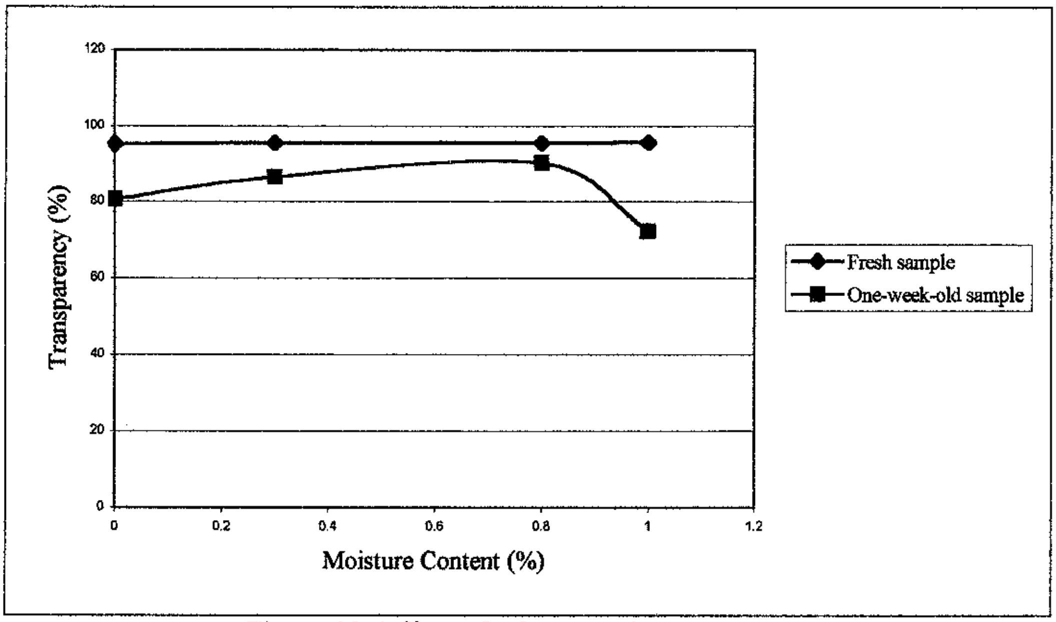


Figure 12: Effect of LOD on gel transparency.

4.2.3 Effect of Heating Temperature on Air Freshener Transparency

The effect of heating temperature on gel transparency is determined by heating the D-Limonene at increasing temperatures. The viscosity of a gel solution decreases with temperature[10]. This explains why the solutions prepared at higher temperatures took a longer time to gel. From Table 5 and Figure 13, it can be seen that the air freshener transparency decreases slightly with increasing temperature. Even after one week, the gel prepared at the highest temperature is the least transparent. As mentioned in the observation, the gel has fewer bubbles at high temperatures. A similar statement in Ref. 11 confirms this observation.

Table 5: Transparencies of air freshener produced with varying temperatures.

Heating Temperature (°C)	Transparency	
	Fresh sample	One-week-old sample
50	97.35	86.38
70	95.60	72.35
90	94.81	82.12
110	93.84	79.40

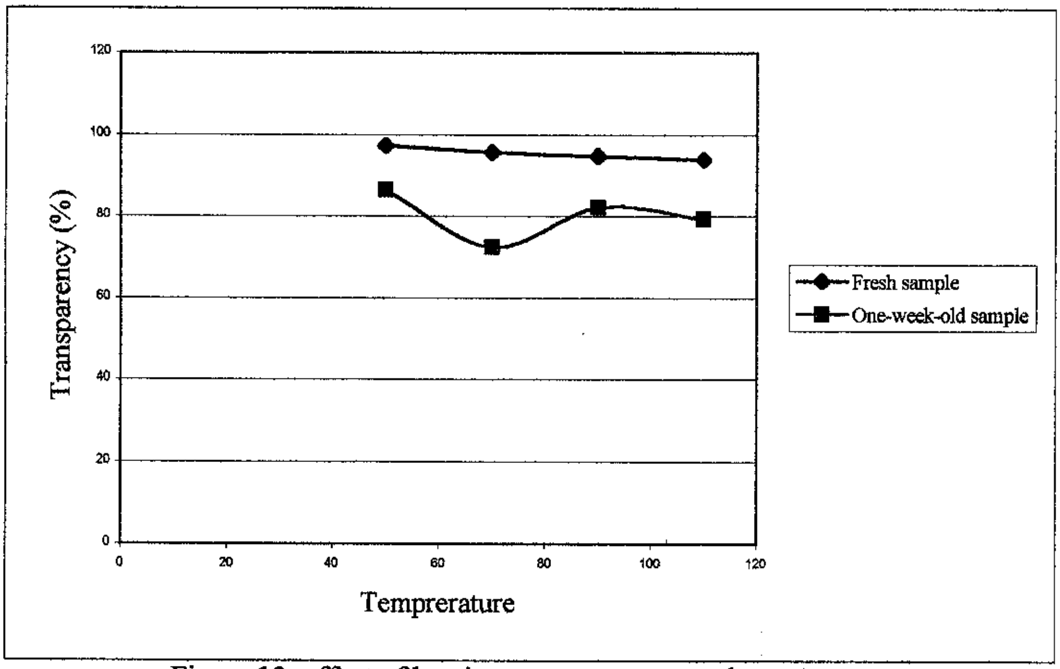


Figure 13: effect of heating temperature on gel transparency.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 RECOMMENDATIONS

There are several factors that a gel-based air freshener manufacturer must take into consideration when producing air freshener. As established by the above discussion, the NaSt produced is capable of performing as a gelling agent. However, the gel produced will not provide satisfactory clarity if other parameters are not kept in check. To ensure that a clear and translucent gel is produced every time, the following parameters should be taken into account:

i. The working area for producing gel air freshener is kept free of contaminants and impurities.
ii. The containers used for storing gel air fresheners are kept dry.
iii. The colouring used to produce the finished air freshener must be chosen carefully. Oil soluble colours are preferable to food colourings[12].
iv. The type and amount of fragrance used in the air freshener is one of the major causes of cloudiness. Only fragrance oils should be used and the amount should not exceed 5%[12].
v. Sucrose (sugar) can be used to improve the clarity of gels[13].

5.2 CONCLUSION

From this project, the student has managed to establish the effects of FFA value, moisture content and heating temperature on gel-based air freshener transparency.

It has been observed that high FFA values cause gel transparency to decrease. However, This observed decrease is only about 2% and the transparency of the gel produced using NaSt with high FFA value is still acceptable. Therefore, it can be concluded that although gel transparency decreases slightly with increasing FFA values, this is not a cause for concern since the maximum allowable FFA value at Stabilchem is 2% for NaSt.

It has also been observed that gel-based air freshener transparency decreases with decreasing NaSt moisture contents. Again, it must be stresses that this decrease is very slight. As can be seen form the graph obtained, the curve is almost a flat line. Therefore, it can be concluded that the effect of NaSt moisture content on air freshener transparency is negligible.

For the effect of heating temperature on gel transparency, it has been observed that transparency decreases with increasing heating temperatures. This decrease in transparency is about 4%, which is quite significant. Therefore, it can be said that air freshener solution should not be prepared at temperatures exceeding 90°C. Another reason why air freshener should not be prepared beyond this temperature is that its viscosity will decrease, thus leading to a longer gelling period.

All in all, it can be summarized that the NaSt 200 and NaSt CP (grinded to 100 mesh) produced in Stabilchem (M) Sdn. Bhd. are suitable to be used in air fresheners. NaSt produced according to the specifications set by the QC are capable of delivering satisfactory clarity upon production. However, the air fresheners produced using these NaSt tend to deteriorate in terms of clarity and shape after one week.

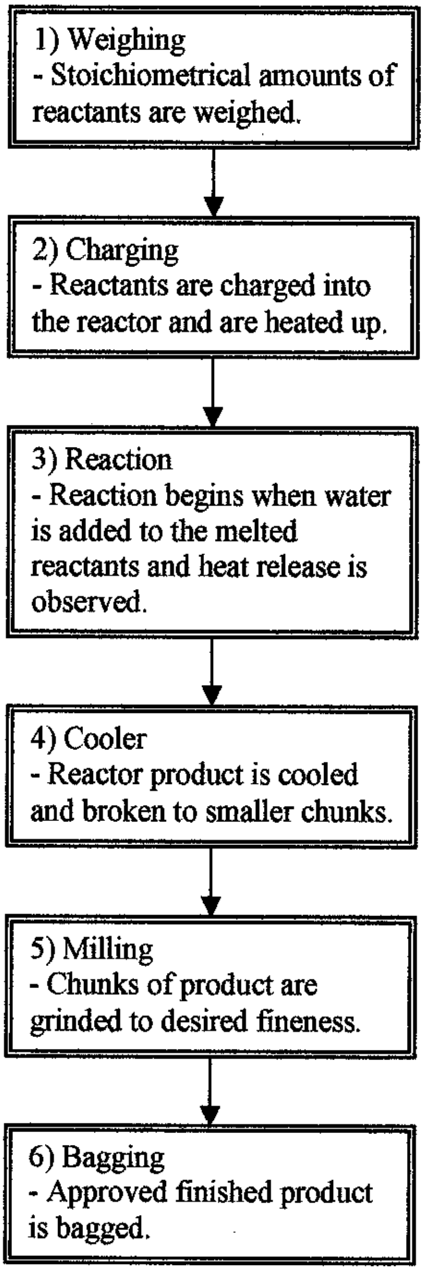
It must be stated that sometimes gels become cloudy because of external factors such as the presence of impurities and moisture. Therefore, care must be taken to ensure that gel air fresheners are produced in a clean and dry environment using compatible additives.

REFERRENCES

1) www.floridachemical.com
2) www.mindfully.org
3) scifun.chem.wisc.edu
4) www.sodiumstearate.com
5) www.iic.co.th
6) Lewis, R. J. Sr., Hawley's Condensed Chemical Dictionary, 14th edition, Wiley, Interscience.
7) www.sbu.ac.uk
8) www.shsu.edu
9) http://oregonstate.edu/instruct/nfm236/starch/index.cfm
10) http://www.geltech.co.kr/htm/products.htm
11) http://www.angelfire.com/sc/candlemaker/Gel-Wax-Info.html
12) http://www.chemistrystore.com/Candle_Gel.htm
13) http://www.candlesupply.com/bathdictionary.html

Appendix I

Process flow diagram for production of NaSt at Stabilchem (M) Sdn. Bhd.



Appendix II

Experimental Methods

Determination of Free Fatty Acid/ Free Alkalinity of Sodium Soaps

Principle

Excess Fatty Acid or Sodium Hydroxide is extracted from the product using Ethanol. The percentage of excess acid or alkali is determined via titration with Sodium Hydroxide (NaOH) or Hydrochloric Acid (HCl).

Apparatus

1. 250ml Erlenmeyer flask.
2. Burette.
3. Analytical balance.
4. Reflux condenser.

Reagents

1. 0.1N HCl.
2. 0.1N NaOH.
3. Neutralised 95% Ethanol.
4. Phenolphthalein indicator.

Procedure

1. 2 g of sample is accurately weighed into a 250ml erlenmeyer flask.
2. 100ml of neutralised 95% Ethanol is added.
3. The solution is refluxed until clear.
4. A few drops of Phenolphthalein are added. If the solution remains clear, it is titrated with 0.1N NaOH. If the solution turns pink, it is titrated with 0.1N HCl.

Calculation

$$\text{Free Fatty Acid (\%)} = \frac{V \times N \times MW}{W \times 10}$$

Where, V = volume of NaOH used for titration.
N = normality of NaOH used.
MW = molecular weight of fatty acid.
= 271
W = sample weight.

$$\text{Free Alkali (\%)} = \frac{V \times N \times 4.0}{W}$$

Where, V = volume of HCl used for titration.
N = normality of HCl used.
W = sample weight.

Determination of Moisture Content/ Loss On Drying

Principle

Loss on drying of Zinc Stearate is determined by radiating the sample with infra-red light.

Apparatus

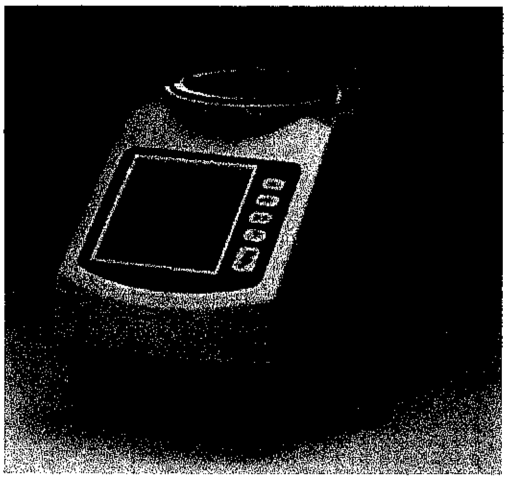
1. Infra-red moisture balance.
2. Aluminuim foil.

Procedure

1. Approximately 3 grams of ZnSt is weighed into an aluminium foil and placed on the weighing pan of the moisture balance.
2. The temperature is set to 100°C for 20 minutes.
3. The result is read of the display after 20 minutes.

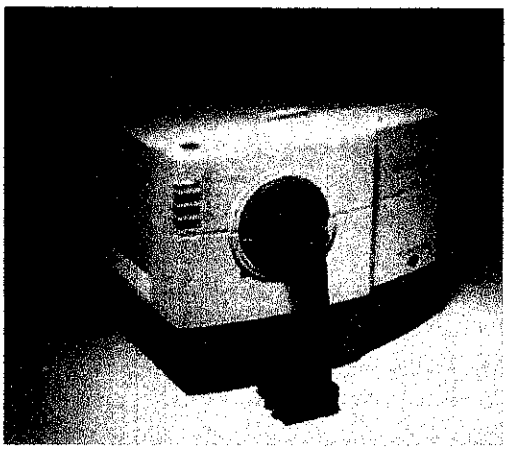
Appendix III

Examples of Hunter Lab Meters



Colour Flex

Lab Scan XE



Ultra Scan XE

Appendix IV

Experimental Data for sample analysis.

Data for Free Fatty Acid value determination of NaSt 200.

Lot Number	Pallet number	Sample weight (g)	Volume of NaOH (ml)	
			Initial	Final
ADC 0057	#7	2.0003	2.55	3.2
		2.0001	15.65	16.45
ADC 0057	#21	2.0003	10.5	10.89
		2.0005	17.4	17.7
ADC 0058	#10	2.0001	10.95	11.57
		2.0004	16.65	17.4
ADC 0058	#12	2.0001	12.15	12.49
		2.0005	16.2	16.65
ADC 0058	#18	2.0010	12.9	13.17
		2.0000	17.7	18.01
ADC 0058	#19	1.9999	13.4	14.28
		2.0003	18.45	19.55

Data for Free Fatty Acid value determination of NaSt CP.

Lot Number	Pallet number	Sample weight (g)	Volume of NaOH (ml)	
			Initial	Final
ADC 0055	#12	2.0001	10.6	11.4
		2.0002	14.5	15.3
ADC 0055	#14	2.0002	11.4	12.04
		1.9999	13.45	14.02

Appendix V

Experimental Data for Transparency Test.

Data for blank petri dish measurement.

Petri dish no.	Measurement 1	Measurement 2	Average
1	0	0.02	0.01
2	0.02	0.08	0.05
3	0.22	0.14	0.18
4	0.11	0.19	0.15
5	0.10	0.18	0.14
6	0.05	0.14	0.10
7	0.07	0.14	0.10
8	0.01	0.06	0.04
9	0.07	0.12	0.10
10	0.15	0.13	0.14
11	0.12	0.15	0.14
12	0.08	0.06	0.07
13	0.05	0.03	0.04
14	0.15	0.12	0.14
15	0.06	0.06	0.06
16	0.10	0.11	0.11

Data for air freshener transparency measurement (NaSt 200)

Lot no.	Pellet no.	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
ADC0057	7	1	7.34	7.62	6.69	6.76	7.10
		2	6.30	6.15	6.44	6.31	6.30
ADC0057	21	3	9.33	9.75	9.58	9.33	9.50
		4	9.01	8.43	8.56	8.44	8.61
ADC0058	10	5	10.23	10.20	9.53	10.69	10.16
		6	9.31	9.53	8.79	9.10	9.18
ADC0058	12	7	9.64	9.93	9.38	10.03	9.75
		8	8.96	9.05	9.88	9.64	9.38
ADC0058	18	9	4.97	5.28	4.94	5.08	5.07
		10	3.71	3.93	4.15	4.10	3.97
ADC0058	19	11	5.72	6.10	6.03	6.08	5.98
		12	8.03	8.70	9.01	8.30	8.51

Data for one-week-old air freshener transparency measurement (NaSt 200)

Lot no.	Pellet no.	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
ADC0057	7	1	25.42	32.65	35.64	27.48	23.43
		2	46.51	46.11	46.05	46.16	46.21
ADC0057	21	3	48.30	47.27	47.92	47.82	47.83
		4	48.54	48.21	47.33	47.99	48.02
ADC0058	10	5	16.58	18.53	22.98	28.03	21.53
		6	50.47	51.62	50.43	50.48	50.75
ADC0058	12	7	25.80	27.89	20.03	24.56	24.57
		8	44.97	45.31	46.19	44.75	45.31
ADC0058	18	9	46.18	45.88	45.74	46.22	46.01
		10	10.39	10.30	7.47	10.01	9.54
ADC0058	19	11	41.25	41.98	42.80	42.78	42.20
		12	46.82	47.29	47.86	47.23	47.30

Data for air freshener transparency measurement (NaSt CP)

Lot no.	Pellet no.	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
ADC0055	12	13	16.34	16.94	17.13	16.43	16.71
		14	8.68	8.74	8.57	8.83	8.71
ADC0057	14	15	6.59	7.21	7.44	7.59	7.21
		16	8.21	8.96	8.82	8.85	8.71

Data for one-week-old air freshener transparency measurement (NaSt CP)

Lot no.	Pellet no.	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
ADC0055	12	13	17.01	17.89	15.87	15.98	16.69
		14	10.08	10.32	10.29	10.24	10.23
ADC0057	14	15	10.22	10.55	10.49	10.21	10.37
		16	13.73	16.53	14.83	14.33	14.86

Appendix VI

Experimental Data for Transparency Test (effect of FFA value).

Data for blank petri dish measurement.

Petri dish no.	Measurement 1	Measurement 2	Average
17	1.25	1.49	1.37
18	0.80	0.66	0.73
19	1.63	1.47	1.55
20	0.48	0.72	0.6
21	1.87	1.83	1.85
22	1.41	1.37	1.39

Data for air freshener transparency measurement (#18, ADC0058).

NaSt weight (g)	FA weight (g)	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
4.9505	0.0504	17	5.14	5.08	5.45	4.83	5.13
		18	4.17	4.55	4.23	4.14	4.27
4.8500	0.1500	19	3.38	3.79	3.79	3.62	3.65
		20	4.05	4.14	4.21	4.18	4.15
4.7504	0.2503	21	5.41	6.26	6.11	6.48	6.07
		22	6.79	7.12	6.86	6.93	6.18

Data for one-week-old air freshener measurement (#18, ADC0058).

NaSt weight (g)	FA weight (g)	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
4.9505	0.0504	17	10.68	9.74	10.03	10.59	10.26
		18	9.64	9.91	10.23	10.02	9.95
4.8500	0.1500	19	8.05	8.42	9.11	8.16	8.44
		20	8.86	9.22	9.10	8.67	8.96
4.7504	0.2503	21	11.27	11.06	11.35	11.22	11.23
		22	11.89	11.74	10.98	12.06	11.67

Appendix VII

Experimental Data for Transparency Test (effect of moisture).

Data for blank petri dish measurement.

Petri dish no.	Measurement 1	Measurement 2	Average
23	0.85	0.81	0.83
24	0.72	0.62	0.67
25	0.65	0.59	0.62
26	1.12	1.44	1.28
27	0.88	0.94	0.91
28	0.58	0.58	0.58

Data for air freshener transparency measurement (#18, ADC0058).

Drying time (min)	LOD (%)	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
15	0.8	23	5.16	4.57	4.03	4.12	4.47
		24	5.35	5.07	4.96	5.10	5.12
30	0.3	25	6.02	5.87	6.08	6.11	6.02
		26	5.18	5.03	4.98	4.97	5.04
60	0	27	4.85	5.25	3.60	5.72	4.86
		28	5.88	5.85	6.48	6.62	6.21

Data for one-week-old air freshener measurement (#18, ADC0058).

Drying time (min)	LOD (%)	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
15	0.8	23	10.23	10.36	10.45	10.08	10.28
		24	11.02	10.56	10.36	11.07	10.75
30	0.3	25	13.68	14.02	13.68	14.22	13.90
		26	15.11	15.46	14.79	14.53	14.97
60	0	27	18.98	20.23	20.15	20.24	19.85
		28	19.95	20.05	20.38	20.61	20.25

Appendix VIII

Experimental Data for Transparency Test (effect of heating temperature).

Data for blank petri dish measurement.

Petri dish no.	Measurement 1	Measurement 2	Average
29	0.68	0.66	0.67
30	3.15	3.81	3.48
31	1.58	0.98	1.28
32	0.83	0.83	0.83
33	0.67	0.57	0.62
34	0.85	0.75	0.80

Data for air freshener transparency measurement (#18, ADC0058).

Heating temperature (°C)	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
50	29	5.49	5.42	5.63	5.62	5.54
	30	3.36	3.86	4.15	4.30	3.92
90	31	6.21	6.12	6.02	6.38	6.18
	32	6.10	6.49	6.35	6.29	6.31
110	33	6.30	6.68	7.45	7.39	6.96
	34	6.76	6.71	6.85	6.86	6.80

Data for one-week-old air freshener measurement (#18, ADC0058)

Heating temperature (°C)	Petri dish no.	Msmnt 1	Msmnt 2	Msmnt 3	Msmnt 4	Average
50	29	15.62	15.23	15.49	15.64	15.50
	30	15.72	15.48	16.13	16.22	15.89
90	31	18.10	18.54	18.54	18.36	18.39
	32	19.13	18.83	18.97	19.24	19.04
110	33	21.22	21.48	21.20	21.57	21.37
	34	22.12	20.78	20.42	21.71	21.26