

ALKALINITY AND MINERAL CONCENTRATION ESTIMATION IN COMMERCIAL  
DRINKING WATER

THOMAS F. FREEMAN HONORS COLLEGE

SENIOR THESIS

BY

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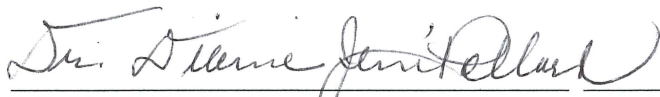
COLLEGE OF SCIENCE, ENGINEERING, AND TECHNOLOGY

MAY 2019

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04/18/2019  
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## ACKNOWLEDGMENTS

I would like to thank God for giving me the opportunities to achieve my goals. I would like to thank Dr. Good for allow me and my colleagues to practice with her. I would like to thank my colleagues, Isoken Idehen, Harold Odili, Stephanie Okoli, and Crista Williams, for helping me complete the research. I would like to thank Dr. Miranda for his valuable support in my thesis. I would also like to thank Dr. Jemison Pollard, Ms. Renuka Nair and Dr. Shandon Neal for their support as well. I would finally like to give my gratitude to my family and close friends. Without their support, none of this would be possible.

## ABSTRACT

### ALKALINITY AND MINERAL CONCENTRATION ESTIMATION IN COMMERCIAL DRINKING

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Several aspects of water and water quality were reviewed and investigated in this study; the effects of dehydration on human health, the role of alkalinity in drinking water, and the actual levels of alkalinity and other minerals in commercial drinking water. Hydration plays a critical role in human physical performance as well as in the prevention of chronic diseases. This experiment was conducted to determine if the level of acidity (or alkalinity) of commercial alkaline water are truly above the normal pH levels, and to identify the salts in each type of water. Based on researches, a more alkaline distilled water has more health benefits in contrast to acidic water, or drinking water with low pH. In this experiment, the mineral content of each water was observed by using the ICPMS. Results showed that the Fiji water sample had the highest amount of calcium, potassium, magnesium and sodium present. This was followed by Alkaline 88, Niagara and lastly, distilled water. Results have shown the positive correlation between mineral content and pH level. Alkaline 88 showed the highest level of alkalinity, with an average pH of 8.26 from the conducted 3 trials. Fiji water had an average pH of 7.02, distilled water had 6.73 and lastly, Niagara had an average pH of 5.42. Niagara, being the cheapest and most popular, had a pH lower than what was put on the label, and the lowest pH of all the samples, which may be 10 to 100 times more acidic than the acceptable values.

# CHAPTER I

## INTRODUCTION

Water is an essential nutrient for life, and hydration plays a critical role in human physical performance as well as in the prevention of chronic diseases. Dehydration is a well accepted contributor to impaired human physical performance, resulting in guidelines established for fluid replacement in many professions involving significant physical activity, including athletes. Performance impairments that are mediated by dehydration can produce untoward effects such as cardiovascular strain, heat strain, altered neurologic function and altered metabolic function (Murray 1996).

With the new wave of certain brands such as Essentia, Fiji, Core etc., the question comes in to play about whether the water is as alkaline as advertised. Of course, the extra few pennies spent on the water could be exchanged for more nutrient-filled water or just for commercial activity. Before answering this question, we must establish what exactly is alkaline water. Alkalinity of water may be due to the presence of one or more of several ions. These include hydroxides ( $\text{OH}^-$ ), carbonates ( $\text{CO}_3^{2-}$ ) and bicarbonates ( $\text{HCO}_3^-$ ). Hydroxide ions are always present in water, even if the concentration is extremely low. Many researches constantly debate on whether an alkaline diet is beneficial or detrimental to the body. But why is alkalinity important? While some say that it helps in regulating the body's pH, others say that it can prevent people from acquiring chronic disease such as cancer (Schwalfenberg 2011). The alkalinity of water can be referred to as "a measure of the capacity of water to neutralize acids". The more acidic the water is, the less capable it is to neutralize acids in the water. This is a measure of pH levels, which determines how acidic or basic (alkaline) water can be. A neutral pH is 7, while those below 7 are acidic. For example, rainwater has a pH of about 5.6 while the

U.S. Environmental Protection Agency (EPA) recommends that public water systems should maintain pH standards of between 6.5 and 8.5. Those above 7 are basic. The alkalinity of water may be defined as its capacity to neutralize acid. Alkali substances in water include hydroxides or bases. Alkaline water companies will typically claim that their water falls between a pH of 8 and 9. Phosphates and silicates are rarely found in natural supplies in concentrations significant in the home. Compounds containing these ions may be used in a variety of water treatment processes.

## REVIEW OF LITERATURE

The history of alkaline water in a sense, came through Michael Faraday, who invented the magneto and dynamo and was one of the great pioneers of electrical energy (A.G.N. 1931). One of his inventions that never achieved any practical use in his lifetime was a device for creating electrolysis, the electronic separation of water into its two principal constituents, hydrogen and oxygen. Later on, a form of electrolysis was used in the creation of the alkaline water ionizer. It would take more than a century for scientists throughout the world to find the inspiration they needed to move forward with their quest to find perfect, healthier water for use by mankind. There were probably a wide variety of people who felt this inspiration. However, the full story of the development of the alkaline water ionizer has yet to be accurately traced.

One of those early researchers, Dr. Henri Coanda, was a renowned Rumanian scientist and Nobel Prize winner. He is known primarily for his study of fluid dynamics and its application to aeronautics. What is less known was his lifelong obsession with the structure of water, an obsession no doubt fueled by the stories of longevity that surfaced regarding the people of Hunza, northern Pakistan (Wilcox et al 2008). As far back as the 1930's, Coanda journeyed to that remote land to confirm his speculations that perhaps the water of Hunza had unique properties that contributed to their longevity. His conclusion was that, indeed, the water was different. When temperature was low, the Hunza water assumed a crystalline, snowflake formation, revealing a structure similar to the venal system in humans, and additionally to the vascular structure in plants. As far as the history of alkaline water ionization goes, all roads lead to Hunza of the Himalayas and to the Andes Mountains, the Shin-Chan areas of China and the

Caucasus in Azerbaijan (Wilcox et al 2008). These remote places are where longevity is the rule, not the exception. The impetus of Hunza water was, and is the common denominator of research into the water of longevity. And since most of the world could not recreate the unusual glacial conditions and mountainous terrain from which this water sprang, scientists looked at the structure of the water to see if it could be created. It was in the early 1960's that the Japanese started research on the possible therapeutic benefits of alkaline ionized water (Tanaka et al 2018). This research was a follow up to earlier work carried out in Russia. The research in Japan focused on using electronic devices to split water into alkaline and acidic streams. As the benefits of alkaline ionized water began to be appreciated, manufacture of water ionizers greatly increased.

During the 1990's manufacturing of water ionizers became more concentrated in Korea, which is today probably the main manufacturing center for these devices. Alkaline ionized water is now commonly drunk in Japan, Korea and many other Oriental countries. It is widely used in North America and now increasingly so in Europe, as awareness has grown about the negative effects of excess acidity from our diets and lifestyles. In the last decade there has been a significant volume of research, particularly in Korea, into the use of special natural minerals to naturally change drinking water into healthy alkaline water. Nowadays there are numerous alkalizing filtration products available. Mineral-based products have several features which make them attractive to consumers. Firstly, they are much more affordable than electronic systems. Secondly, they are more natural, giving you water which in many ways has characteristics like naturally-occurring healing waters. In addition to alkalizing minerals, most of these systems use minerals such as tourmaline which emits far infra-red energy which has healing effects.



*Health Benefits.* As a consumer, you might question whether or not alkaline water is useful for your health benefits. For example, a study found that drinking naturally carbonated artesian-well alkaline water with a pH of 8.8 may help deactivate pepsin, the main enzyme that causes acid reflux (Zalvan et al 2017). At the cellular level, tissue-bound pepsin is fundamental to the pathophysiologic mechanism of reflux disease, and although the thresholds for laryngeal damage in laryngopharyngeal reflux and for esophageal damage in gastroesophageal reflux disease differ, both forms of damage are due to pepsin, which requires acid for its activation. In addition, human pepsin remains stable at pH 7.4 and may be reactivated by hydrogen ions from any source. Thus, most tap and bottled waters (typically pH 6.7 to 7.4) would not be expected to affect pepsin stability. The purposes of these *in vitro* studies were to investigate whether artesian well water containing natural bicarbonate (pH 8.8) might irreversibly denature (inactivate) human pepsin, and to establish its potential acid-buffering capacity. Those who consumed high pH water reduced viscosity by 6.3 percent compared to 3.36 percent with standard purified drinking water. This means blood flowed more efficiently with alkaline water. This can increase oxygen delivery throughout out the body. Laboratory studies were performed to determine whether human pepsin was inactivated by pH 8.8 alkaline water. In addition, the buffering capacity of the alkaline water was measured and compared to that of the two most popular commercially available bottled waters. The pH 8.8 alkaline water irreversibly inactivated human pepsin (in vitro), and its hydrochloric acid–buffering capacity far exceeded that of the conventional-pH waters. Unlike conventional drinking water, pH 8.8 alkaline water instantly denatures pepsin, rendering it permanently inactive. In addition, it has good acid-buffering capacity. Thus, the consumption of alkaline water may have therapeutic benefits for patients with reflux disease.

Another study shows that drinking alkaline ionized water may have benefits for people who have high blood pressure, diabetes, and high cholesterol. A study conducted in China was conducted to observe the changes of blood pressure, blood sugar and blood lipids after alkaline ionized drinking water was used in cases of essential hypertension, diabetes mellitus (consists of mild degree of increased blood sugar) and hyperlipidaemia (Jackson et al 2018). The experiment was conducted by assessing the subjects Blood pressure, blood sugar and blood lipids initially before the study-and then 3 to 6 months after alkaline ionized water was drunk. The levels of blood pressure, blood sugar and blood lipids decreased significantly even to normal ranges after alkaline ionized water was drunk. Researches then concluded that alkaline ionized water may be used as one of the accessory therapeutic methods for essential hypertension, diabetes mellitus and hyperlipidaemia. However, the scientists point out that participants had different measures of whole blood viscosity at the outset, which could have affected the results. It is worth noting that the study was sponsored by Essentia Water, who also provided the alkaline water used.

More recent studies were conducted in New Jersey over the same assessment of whether alkaline water effects blood viscosity after consuming it for exercise (Chyki et al. 2018, Weidman et al. 2016). This study, performed at the Waterfront Technology Center was a randomized, double-blind, parallel-arm, controlled trial, which recruited 100 adult volunteers (50 male, 50 female), between 25 to 49 years of age. Eligible participants were healthy, non-smoking adults, having a body-mass index less of 29 or less and free from any medication for at least one week prior to the participation in the study. Female participants were excluded from the study if they were pregnant, breast-feeding, menstruating at the time of screening, or if they had taken oral contraceptives in the previous 3 months. Subjects were instructed to refrain from strenuous activity, alcohol, and to limit excessive caffeine intake (>2 six-ounce cups) for at least 24 h prior

to their assigned arrival on the study date. This clinical study was approved by the Institutional Review Board, and written informed consent was obtained from all subjects at the time of enrollment and prior to participating in this study. The study was registered (ClinicalTrials.gov Identifier: NCT02118883) and conducted in accordance and compliance with Good Clinical Practice and the Declaration of Helsinki. Subjects were permitted to consume food and water at will prior to the study. Following a baseline assessment, participants were asked to refrain from food or fluid intake. Baseline assessments for body mass, bioelectrical impedance and vital signs (heart rate (HR), systolic (SBP) and diastolic blood pressure (DBP), respiration rate, body temperature) were collected at the initiation of the study prior to exercise. Blood samples were collected by venipuncture for evaluation of whole blood viscosity and plasma osmolality. Following baseline measures, the subjects performed moderate aerobic exercise sessions (using their choice of a treadmill, stationary bicycle, and/or elliptical trainer) in a warm environment (30 °C, 70% relative humidity) (Weidman et al. 2016). The duration of exercise varied between subjects; however, the dehydration threshold target was standardized to  $2.0 \pm 0.2\%$  body weight loss due to the effects of a period of exercise in producing mild dehydration. During the exercise period, participants dried themselves thoroughly before each body mass measurement. A disposable paper gown of known weight was provided during body mass measurements. After the exercise period was completed and a dehydrated state attained, study participants moved to a thermo-neutral environment (21 °C, 60% relative humidity), where they rested for 20 min. After this rest period, vital signs, weight and bioimpedance were assessed. In addition, blood samples were collected for assessment of blood viscosity and plasma osmolality. A prior study, assessing the effect of oral carbohydrate solution on rates of absorption reported an approximate 3% reduction in plasma volume during a 105-min interval after beverage consumption (Jeukendrup

2014). The present study incorporated a follow-up period of 120 min, which was considered to be sufficiently long in duration to show any effect of rehydration during recovery. The 120-min follow-up period (T000 to T120 min), which followed exercise and rest, was divided into a 30-min rehydration period and a 90-min recovery period. Participants were rehydrated orally by CON or ALK (T000 to T030 min). The mass of the water consumed during the rehydration period was calculated according to a participant's body mass change during the exercise period. The recommended amount of rehydration fluids was determined using a formula of 20 mL of oral hydration per 1 kg of subject body weight, i.e. 2% of pre-exercise, baseline body weight. Water volumes poured into containers were measured using a precision scale (Intelligent-Lab PD-3000, Intelligent Weighing Technology, Inc. Camarillo, CA) by an unblinded coordinator who had no contact with any participants or study results throughout the study. Subjects were required to consume the entire quantity of designated water following exercise ad libitum within 30 min (T000 to T030 min). Blood samples were collected for whole blood viscosity and plasma osmolality at T015 min and T030 min during this rehydration period. The scanning capillary viscometer used to assess the primary endpoint in this study was previously employed in a preliminary study of dehydration and rehydration by high pH alkaline water in 15 nonsmoking, apparently healthy firefighters. The variability of systolic blood viscosity measurements (high-shear viscosity) and the rehydration effect of high-pH alkaline on systolic viscosity observed in this prior study population were used to determine the sample size for this study [12]. In this firefighter trial, dehydration induced by fighting mock fires in training session with full equipment produced mean systolic viscosity values of 42.7 mP, and after rehydration, mean systolic blood viscosity was significantly reduced to 38.8 mP ( $p=0.003$ ). A standard deviation of 2.6 mP observed at baseline was used in determining our sample size for the present study. We

postulated that high-pH ALK would demonstrate 40% greater rehydration effect than CON, that is, rehydration by CON was hypothesized to reduce mean systolic blood viscosity to 40.5 mP while ALK was hypothesized to reduce systolic blood viscosity to 38.8 mP from a dehydrated level of 42.7 mP. The present study was powered to detect such a contrast with 90% power using a type I error rate of 5%. This required 100 participants or 50 in the CON group and 50 individuals in the ALK group.

Statistical analyses were performed using SAS (Statistical Analysis System, Version 9.3, 2012, Cary, NC). The data were analyzed using both descriptive and inferential statistics. Four separate analyses were pre-planned: comparison of percent change in biomarkers, comparison of the slopes of regression lines, absolute differences, and mixed model analyses. Mean values for each treatment group and estimates of standard errors for each enabled confidence intervals were to be computed and conclusions made based on these differences. Results can be viewed below (Figure 1).

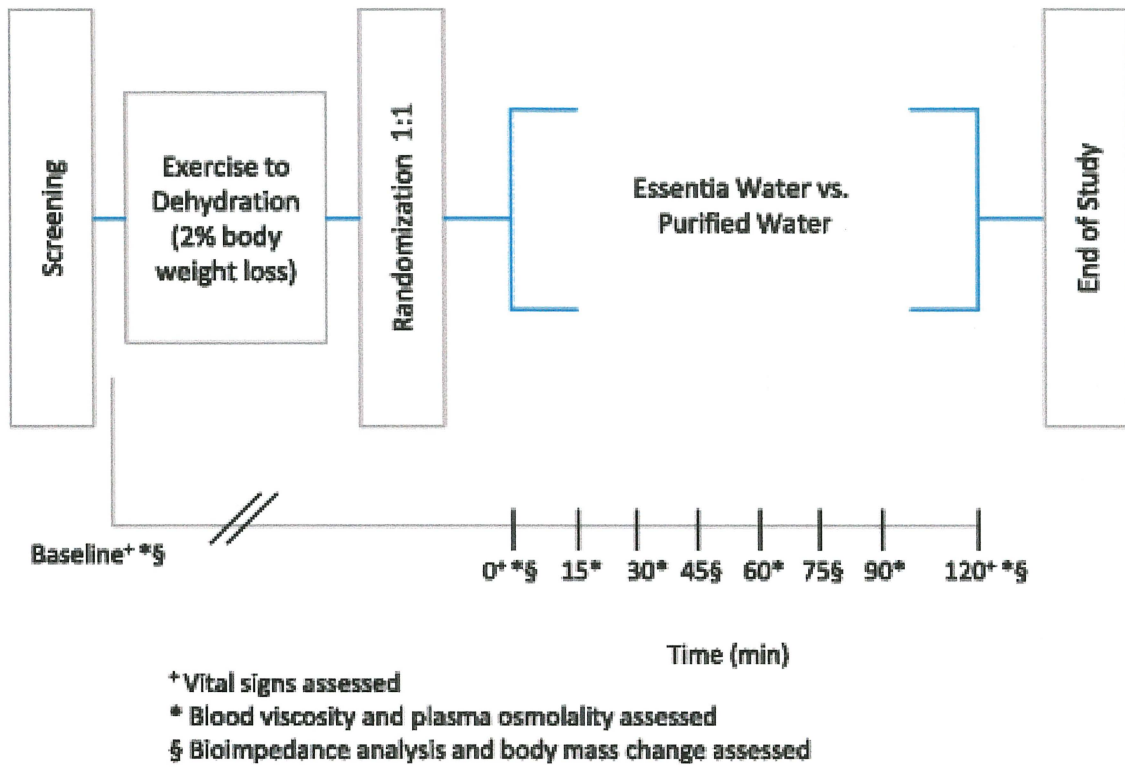


Figure 1: Clinical Study Flow Sheet

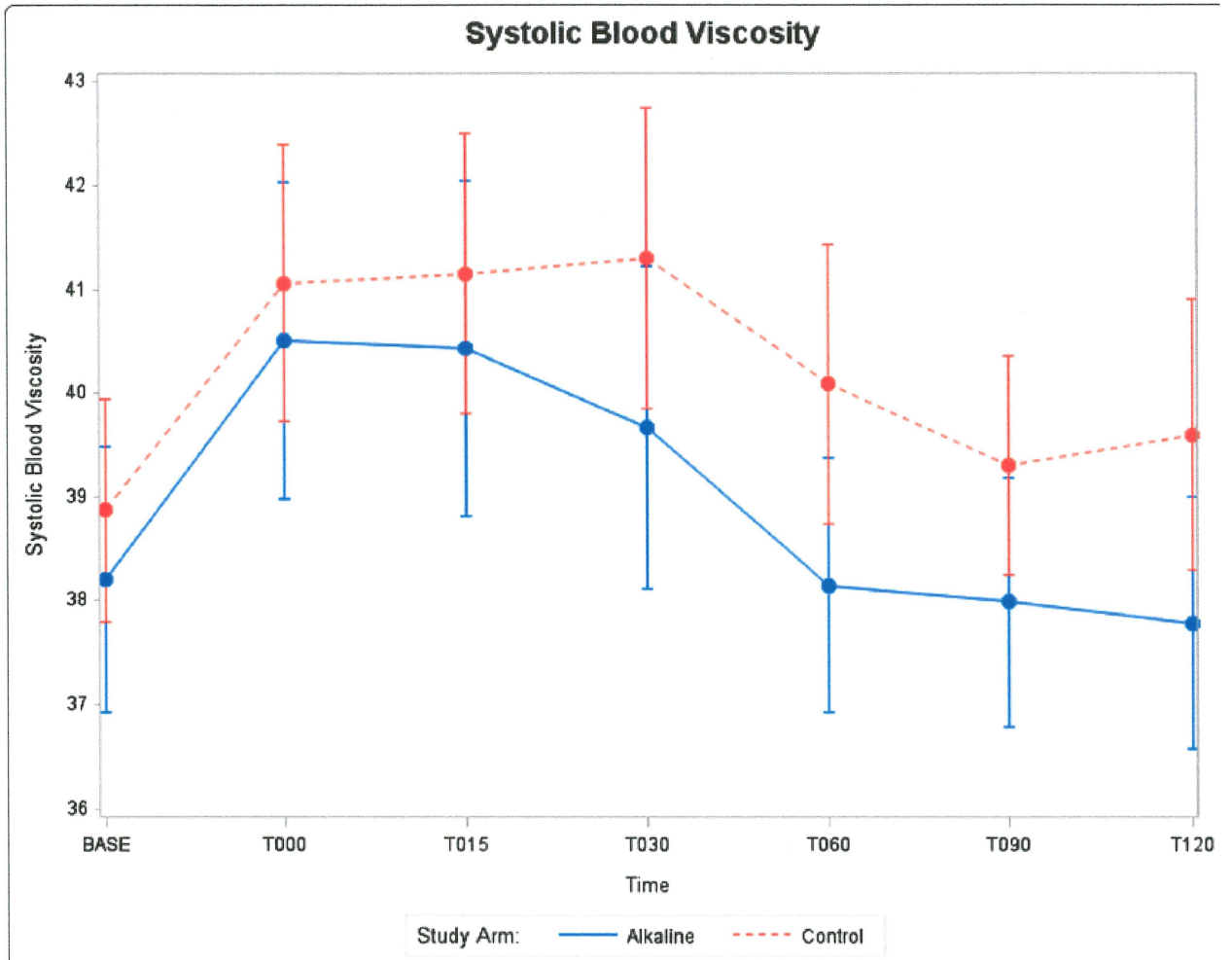


Figure 2: Systolic blood viscosity

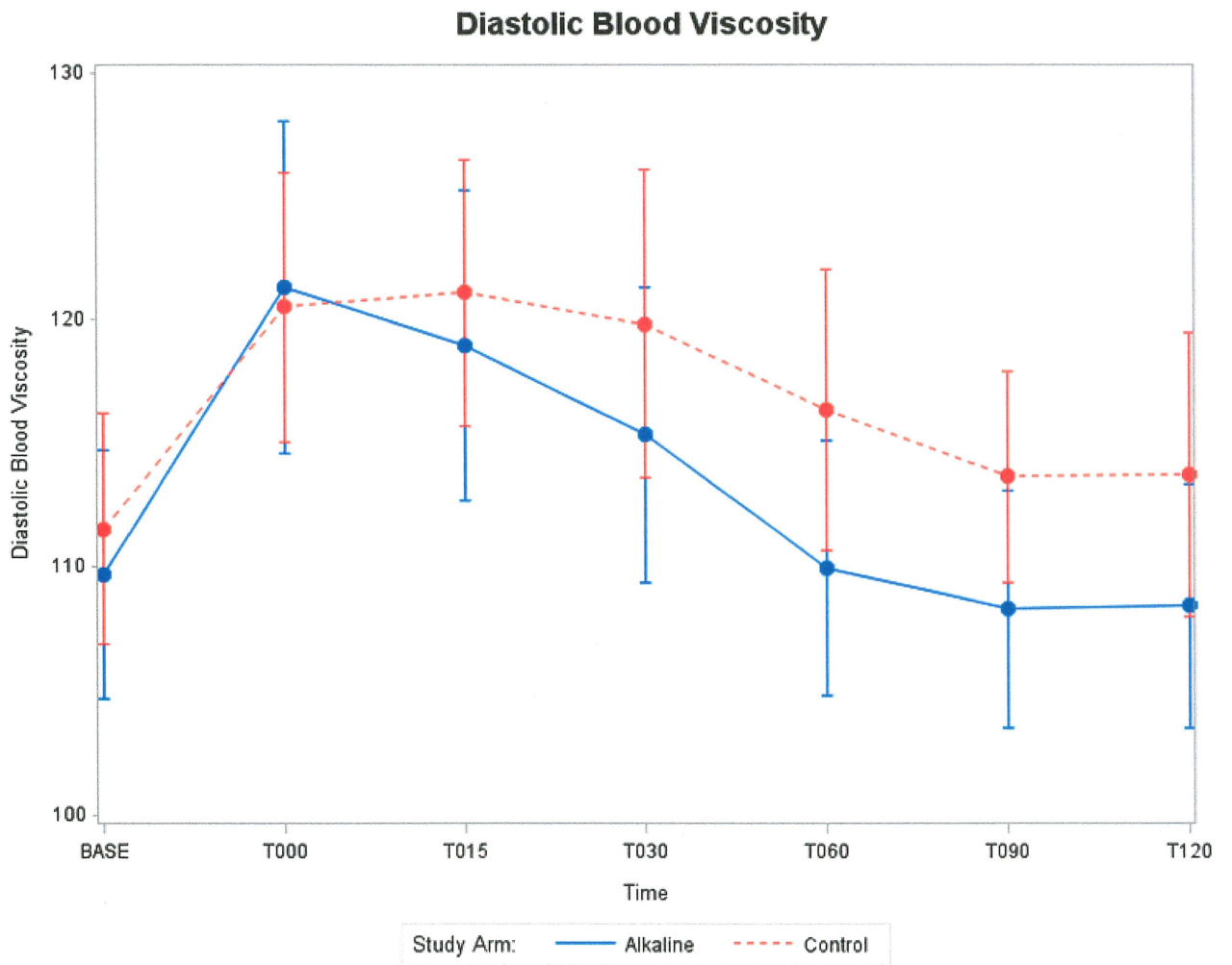


Figure 3: Diastolic blood viscosity



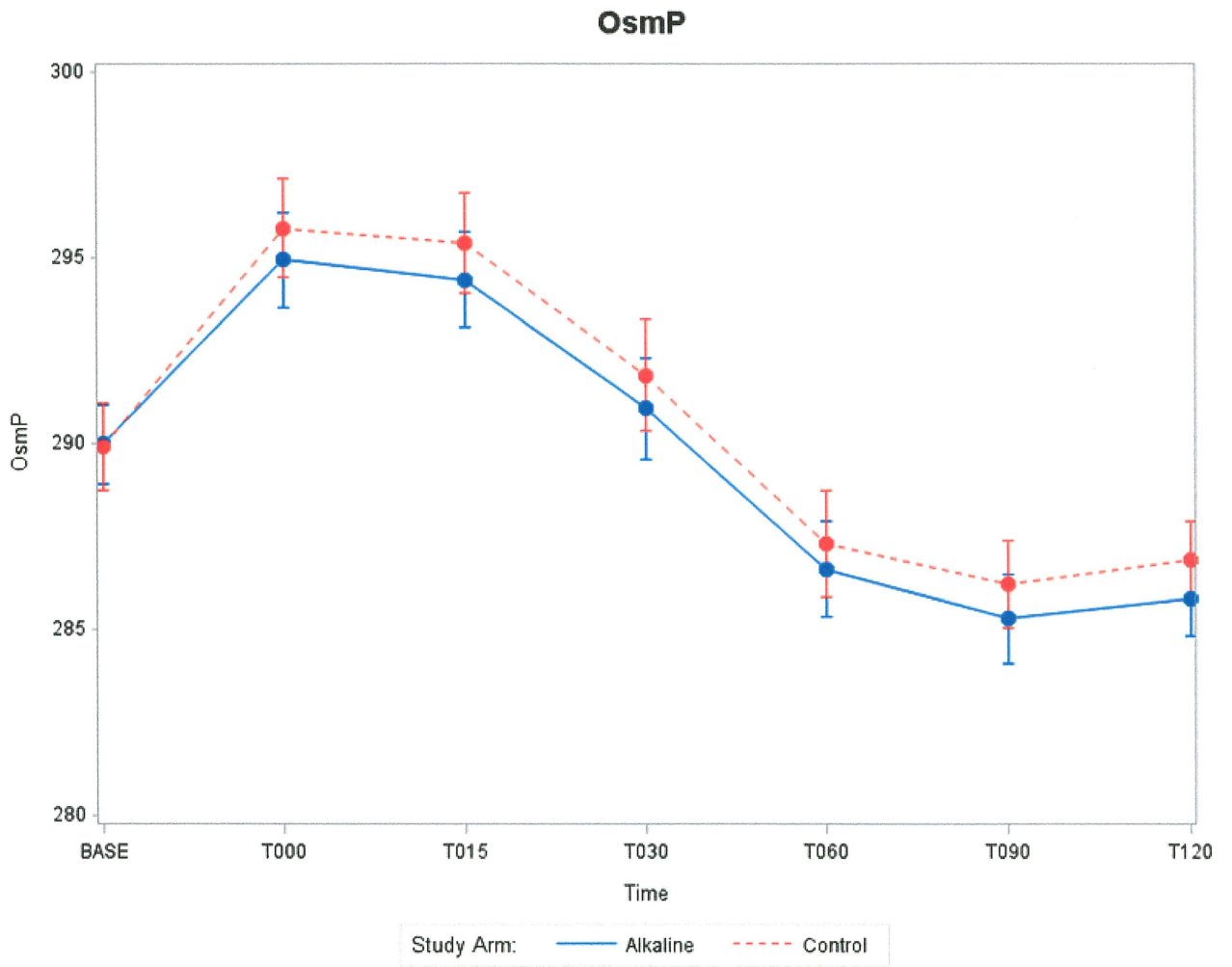


Figure 5: Plasma osmolality

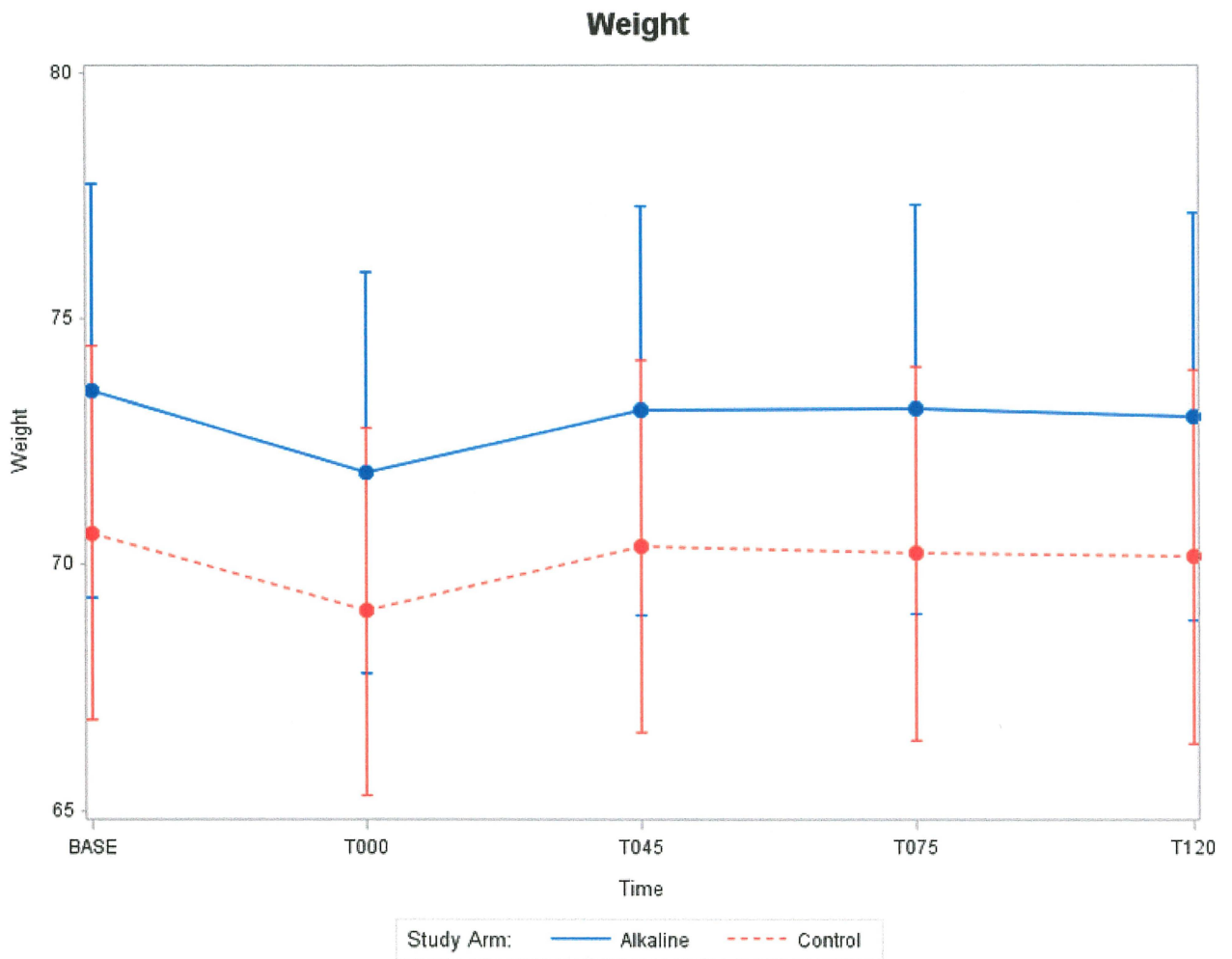


Figure 6: Weight of Subjects.

This randomized, double-blinded, parallel-arm controlled study compared the rehydration effect of ALK to CON in order to characterize relative hydration efficacy and performance. A pre-planned analysis of percentage changes, starting at dehydration (T000) and ending at recovery (T120), enabled the two treatment groups to be compared while reducing the impact of

inter-individual variability. For systolic blood viscosity, ALK demonstrated significantly greater rehydration than CON ( $p = 0.03$ ), and this result was consistent with the findings using the mixed model analyses. Separately, a study using an exercise-induced dehydration protocol to compare the effect of two fluid replacement beverages on markers for oxidative stress showed that rehydration recovery following ingestion of either a carbohydrate-electrolyte beverage or water reduced levels of malondialdehyde, a common marker for oxidative stress, relative to plasma concentrations of malondialdehyde at a dehydrated state. Disruptions in blood flow promote an oxidative state where reactive oxygen species accumulate. Red blood cells in particular are vulnerable to an oxidative environment in the human body and, as a consequence of their iron content, are capable of producing their own free radicals. This process of autoxidation occurs when oxygenated hemoglobin is degraded and releases a superoxide. Concurrently, the ferrous ( $Fe^{2+}$ ) state iron in hemoglobin is oxidized to ferric ( $Fe^{3+}$ ) hemoglobin, producing methemoglobin which is incapable of transporting oxygen.

In conclusion with this study, it was designed to characterize differences between ALK and CON in terms of intravascular hydration as quantified by serial changes in systolic blood viscosity following exercise-induced dehydration. Those who consumed high pH water reduced viscosity by 6.3 percent compared to 3.36 percent with standard purified drinking water. This means blood flowed more efficiently with alkaline water. This can increase oxygen delivery throughout the body. Drinking high-pH ALK was shown to reduce systolic blood viscosity significantly more than CON consumption following exercise-induced dehydration, when comparing the percent change in WBV from a dehydrated state to 120 min after rehydration and recovery. A mixed model analyses validated this significant treatment effect for high-pH ALK on systolic blood viscosity vs. CON. Absolute differences at multiple time points did not

demonstrate any significant differences; however, the subjective observed benefit may be attributed to the high variability of WBV measurements in the study groups.

Moderate concentrations of alkalinity are desirable in most water supplies to balance the corrosive effects of acidity. However, excessive quantities cause a number of problems. These ions are, of course, free in the water, but have their counterpart in cations such as calcium, magnesium and sodium or potassium. You probably will not notice an alkaline condition due to bicarbonate ions except when present in large amounts. In contrast, you should readily detect alkalinity due even to fairly small amounts of carbonate and hydroxide ions. An article, 'The Alkaline Diet: Is There Evidence That an Alkaline pH Diet Benefits Health?' written by Gerry K. Schwalfenberg, states that "Human life requires a tightly controlled pH level in the serum of about 7.4 (a slightly alkaline range of 7.35 to 7.45) to survive." However, because modern diet significantly differs from the hunter's civilization, there has been a tremendous decrease in potassium and a great increase in sodium and chloride, making diets less and less alkaline. It is now generally accepted to have a potassium and magnesium deficient diet.

Despite many research studies conducted on alkaline water, there needs to be more studies conducted to further prove its health benefits. However, there are other questions that are raised for alkaline water.

*Health Risks.* Of course, with there's good in the enrichment of alkaline water, there are of course side effects. Some examples of negative side effects include the lowering of natural stomach acidity, which helps kill bacteria and expel other undesirable pathogens from entering your bloodstream. Additionally, an overall excess of alkalinity in the body may cause gastrointestinal issues and skin irritations. Too much alkalinity may also agitate the body's

normal pH, leading to metabolic alkalosis, a condition that may produce the following symptoms: nausea, vomiting, hand tremors, muscle twitching, tingling in the extremities or face and/or confusion. Alkalosis can also cause a decrease in free calcium in the body, which can affect bone health. However, the most common cause of hypocalcemia isn't from drinking alkaline water, but from having an underactive parathyroid gland.

### *Is Alkaline Water Natural or Artificial?*

Of course, as a consumer, you may wonder if commercialized alkaline water is made naturally or artificially. Alkaline Water comes in 2 different forms, natural alkaline water, which is naturally alkaline from the source and requires no additional processing to reach a high pH. An example is our own PH8 Natural Alkaline Water which obtains its alkalinity and essential mineral content when filtering down to the aquifer through thick black limestone. Artificial alkaline water on the other hand is produced when neutral or tap water has been transformed by an electronic process. Artificially produced alkaline water does not contain naturally occurring minerals, however, many products use added minerals to make it seem more natural. Because on the bottle itself it's hard to determine what is natural and what is processed. Many bottled water companies and machine-based systems will try to mislead customers by marketing their products as 'natural' despite using electrolysis / water ionizers. This might suggest that commercialized water may have been chemically altered instead of being 100 percent natural as advertised. Despite this, will this affect whether the water is alkaline as advertised? If water companies can falsely advertise that their water is 100 percent natural, what's there to say that the water companies can just advertise that their waters are alkaline but in reality, provide regularly distilled water or even acidic water?

### *Alkaline Water Electrolysis*

As mentioned previously, the alkaline water is produced using the process of electrolysis. Commercialized alkaline water electrolysis has been considered a sustainable approach to producing hydrogen using renewable energy inputs but preventing hydrogen/oxygen mixing and efficiently using the instable renewable energy are challenging. Here, using nickel hydroxide as a redox mediator, we decouple the hydrogen and oxygen production in alkaline water electrolysis, which overcomes the gas-mixing issue and may increase the use of renewable energy. In this architecture, the hydrogen production occurs at the cathode by water reduction, and the anodic  $\text{Ni(OH)}_2$  is simultaneously oxidized into  $\text{NiOOH}$ . The subsequent oxygen production involves a cathodic  $\text{NiOOH}$  reduction ( $\text{NiOOH-Ni(OH)}_2$ ) and an anodic  $\text{OH}^-$  oxidization. Alternatively, the  $\text{NiOOH}$  formed during hydrogen production can be coupled with a zinc anode to form a  $\text{NiOOH-Zn}$  battery, and its discharge product (that is,  $\text{Ni(OH)}_2$ ) can be used to produce hydrogen again. Figure 7 shows the oxygen evolution involved with the process

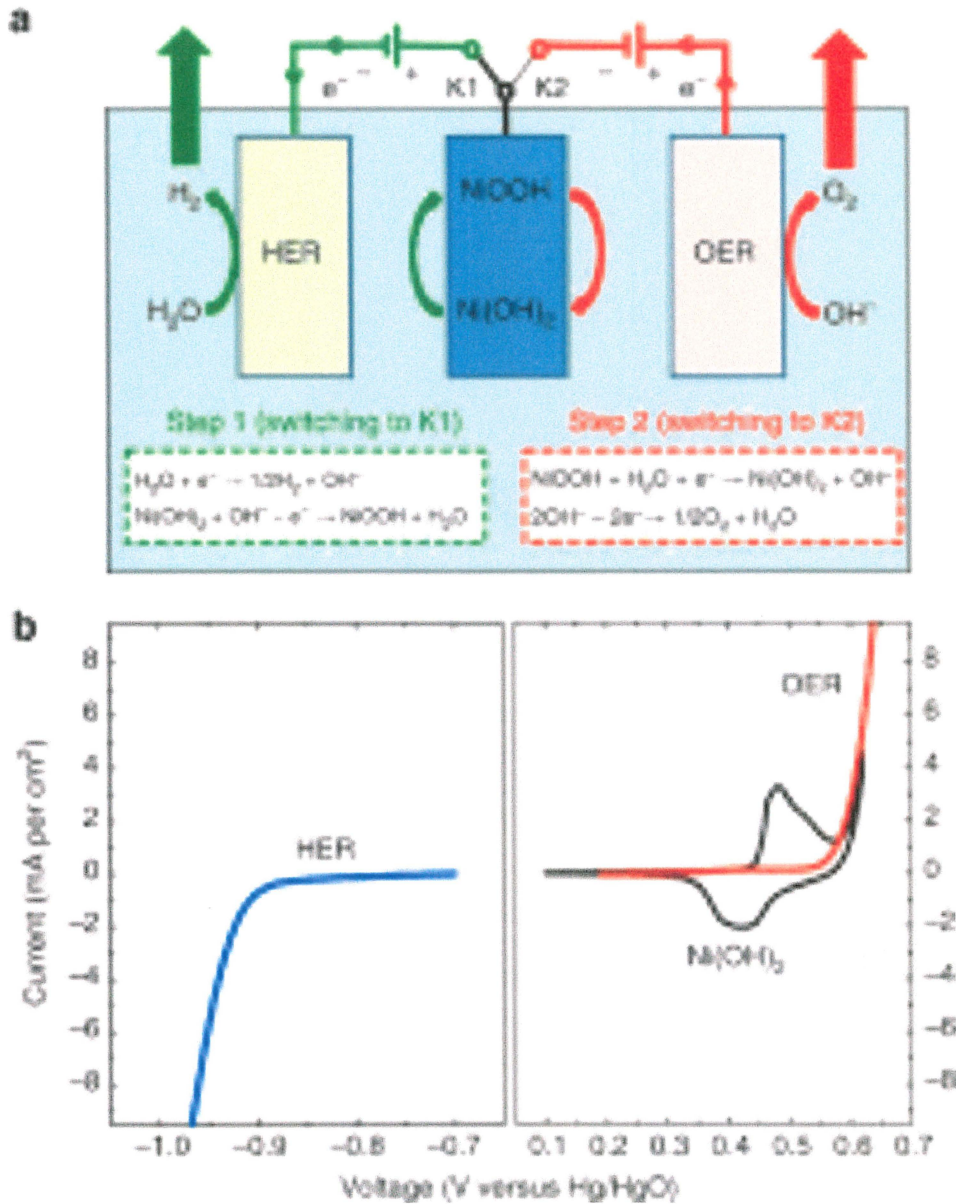


Figure 7: Oxygen evolution in Alkalinity water

As shown in Fig. 7, the H<sub>2</sub> production (Step 1) involves a cathodic reduction of H<sub>2</sub>O on the HER electrode (H<sub>2</sub>O-H<sub>2</sub>) and a simultaneous anodic oxidation of the Ni(OH)<sub>2</sub> electrode (Ni(OH)<sub>2</sub>- NiOOH). The subsequent O<sub>2</sub> production (Step 2) occurs on the OER electrode by an anodic oxidation of OH (OH - O<sub>2</sub>), whereas the NiOOH cathode is reduced to

Ni(OH)<sub>2</sub>. This approach leads to a device architecture for the alkaline electrolytic cell with several important advantages. First, the separate generation of O<sub>2</sub> and H<sub>2</sub> prevents the product gases from mixing over a range of current densities and simplifies the gas handling, which greatly increase the operation flexibility of alkaline electrolytic cells and make them suitable to be driven by sustainable energy (such as solar energy). Second, this device architecture can produce highly pure H<sub>2</sub> and O<sub>2</sub> with nonmembrane, which further reduces the cost of the alkaline water electrolysis technology. Third, the separate H<sub>2</sub> and O<sub>2</sub> productions require different driving voltages (or power inputs), which implies that we can flexibly use sustainable energy (such as solar or wind power) for H<sub>2</sub> production or O<sub>2</sub> production based on the output variation in these unstable power sources. Finally, the NiOOH that forms during the H<sub>2</sub> production (that is, Step 1) can be coupled with a zinc anode to form a NiOOH-Zn battery for energy storage, and its discharge depends on the cathodic reduction of the NiOOH electrode (NiOOH- Ni(OH)<sub>2</sub>) and the anodic oxidization of the zinc electrode (Zn- ZnO<sub>2</sub>). Herein, it should be noted that the cathodic reduction potential of NiOOH (0.45 V versus Hg/HgO) is significantly higher than the anodic oxidization potential of zinc.

Interestingly, the O<sub>2</sub>-production step (Step 2) can be replaced by the discharge step of the NiOOH-Zn battery (Step 20), which will enable the coupling of H<sub>2</sub> production with a discharge step of the NiOOH-Zn battery (Fig. 4a). As shown in Fig. 4a, the H<sub>2</sub> production (Step 1) includes the cathodic reduction of H<sub>2</sub>O on the HER electrode (H<sub>2</sub>O-H<sub>2</sub>) and the anodic oxidization of the Ni(OH)<sub>2</sub> electrode (Ni(OH)<sub>2</sub>-NiOOH). Next, the NiOOH electrode that is formed in Step 1 is coupled with a zinc anode to form a NiOOH-Zn battery. The subsequent discharge step (Step 20) of the NiOOH-Zn battery is based on the cathodic reduction of the NiOOH electrode (NiOOH-Ni(OH)<sub>2</sub>) and the anodic oxidization (Zn-ZnO<sub>2</sub>)



2 ) of zinc<sup>48–50</sup>. In other words, the architecture in Fig. 4a provides an interesting rechargeable cycle that produces H<sub>2</sub> with charge (that is, electrolysis in Step 1) and delivers energy with the discharge of the NiOOH-Zn battery (Step 20). To confirm this hypothesis, the NiOOH electrode, which formed after the electrolysis for H<sub>2</sub> production with an applied current of 200 mA for 600 s, was directly coupled with a zinc-plate electrode in the electrolysis cell to construct a NiOOH-Zn battery. The discharge profile of the NiOOH-Zn battery was investigated with a current of 200 mA. As shown in Fig. 4b, the NiOOH-Zn battery displays a discharge voltage of 1.6 V with a total discharge time of 600 s. The cycle of Steps 1 and 20 also increases the Zn(OH)<sub>2</sub> concentration in the alkaline electrolyte. The Zn(OH)<sub>2</sub> alkaline solution can be used to produce O<sub>2</sub> and metallic Zn through electrolysis at the proper time with other energy inputs, such as at night-time with wind power, nuclear fission and so on. As at night-time with wind power, nuclear fission and so on. In addition, the rechargeable system based on the H<sub>2</sub> production (Step 1) and discharge of the NiOOH-Zn battery (Step 20) exhibits a theoretical energy density of 280 Wh kg<sup>-1</sup> (see Supplementary Fig. 19 for details), which is close to the theoretical energy density of conventional Ni-MH batteries, Ni-Cd batteries or Ni-Zn batteries and higher than the theoretical energy density of lead-acid batteries and aqueous Li-ion batteries<sup>51</sup>.

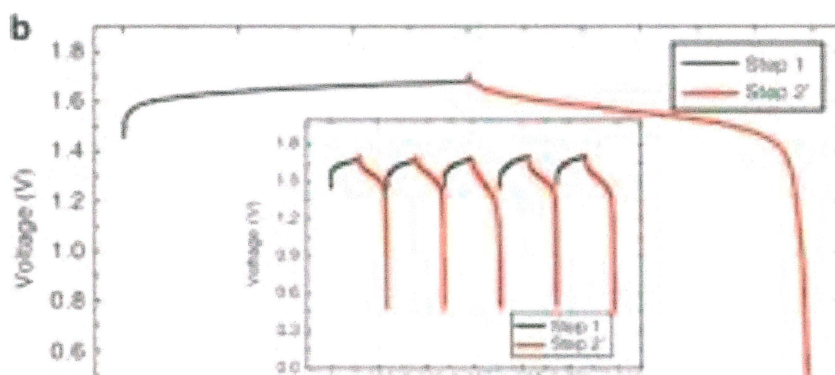
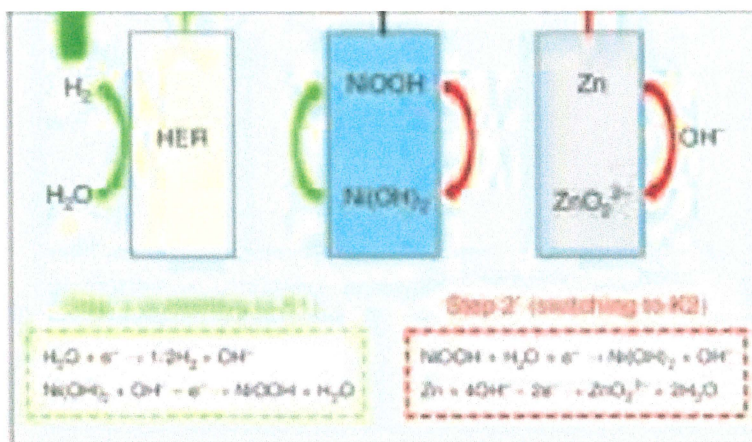


Figure 8: Combination of the H<sub>2</sub>-production and the NiOOH-Zn battery

The separate H<sub>2</sub> and O<sub>2</sub> production overcomes the challenge of H<sub>2</sub>/O<sub>2</sub> mixing and facilitates the operation of alkaline electrolysis even with unstable power inputs. The separate H<sub>2</sub> and O<sub>2</sub> productions require different driving voltages (or input power), which implies that we can flexibly use sustainable energy, such as solar or wind power, with higher efficiency. Finally, the combination of H<sub>2</sub> production and discharge of the NiOOH-Zn battery potentially provides a new energy storage/conversion approach for human life. It should also be noted that the electrochemical redox process of Ni(OH)<sub>2</sub>/NiOOH is generally limited by the proton diffusion in the crystalline framework of Ni(OH)<sub>2</sub> or NiOOH<sup>52</sup>, which limits the electrolysis rate.

### *Objective of Thesis*

Because water is such an essential part of diet, both in the past and now, it is important to know the quality of the water made readily available to the public. This is important because consumers should know what exactly they are buying as well as they are not just buying water for the brand or advertising. In this experiment, popular brands were tested to measure the pH levels and determine if they follow standard recommendations and if their claims of alkalinity were honest. Also, a study was done to identify the different background salts and minerals found in each brand of water. The brands that were tested were Niagara, Fiji, Alkaline 88 and distilled water found in a chemistry lab at Texas Southern University.

## Chapter II

### MATERIALS AND METHODS

The different types of water bottles were purchased at nearby grocery stores. Inductively Coupled Plasma Mass Spectrometry or ICP-MS is an analytical technique used for elemental determinations. The technique was commercially introduced in 1983 and has gained general acceptance in many types of laboratories. Geochemical analysis labs were early adopters of ICP-MS technology because of its superior detection capabilities, particularly for the rare-earth elements (REEs). ICP Analysis, also called ICP Testing, is performed to identify and measure a range of chemical elements necessary for the analysis of metal samples. Both ICP test methods can be performed on solid or liquid samples. They calculate quantitative and qualitative data that can be included in an ICP test report. LTI offers ICP metal analysis services and can determine up to 70 elements using the latest technology in spectrometers at our United States facility in the Philadelphia, PA area. Trace unknowns can be detected and identified. In addition, ICP analysis can reveal several non-metals. Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) or ICP Atomic Emission Spectroscopy is a technique that can determine concentrations of trace to major elements and can detect most elements in the periodic table. Reliable results can be obtained for about 70 elements with detection limits in the parts per billion range. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or ICP Mass Spectrometry is highly sensitive and capable of multi-element trace analysis and ultra-trace analysis, often at the parts-per-trillion level. Testing for trace elements can be performed on a range of materials from super alloys to high purity materials. An example of this ICP-MS machine can be seen by the figure below.



Figure 9: A photograph of the ICP-MS



Below is the Interface of the ICP. The sampler and skimmer cones are metal disks with a small hole (~1mm) in the center. The purpose of these cones is to sample the center portion of the ion beam coming from the ICP torch. A shadow stop (see Figure 2) or similar device blocks the photons coming from the ICP torch, which is also an intense light source. Due to the small diameters of the orifices in the sampler and skimmer cones, ICP-MS has some limitations as to the amount of total dissolved solids in the samples. Generally, it is recommended that samples have no more than 0.2% total dissolved solids (TDS) for best instrument performance and stability. If samples with very high TDS levels are run, the orifices in the cones will eventually become blocked, causing decreased sensitivity and detection capability and requiring the system to be shut down for maintenance. This is why many sample types, including digested soil and rock samples must be diluted before running on the ICP-MS.

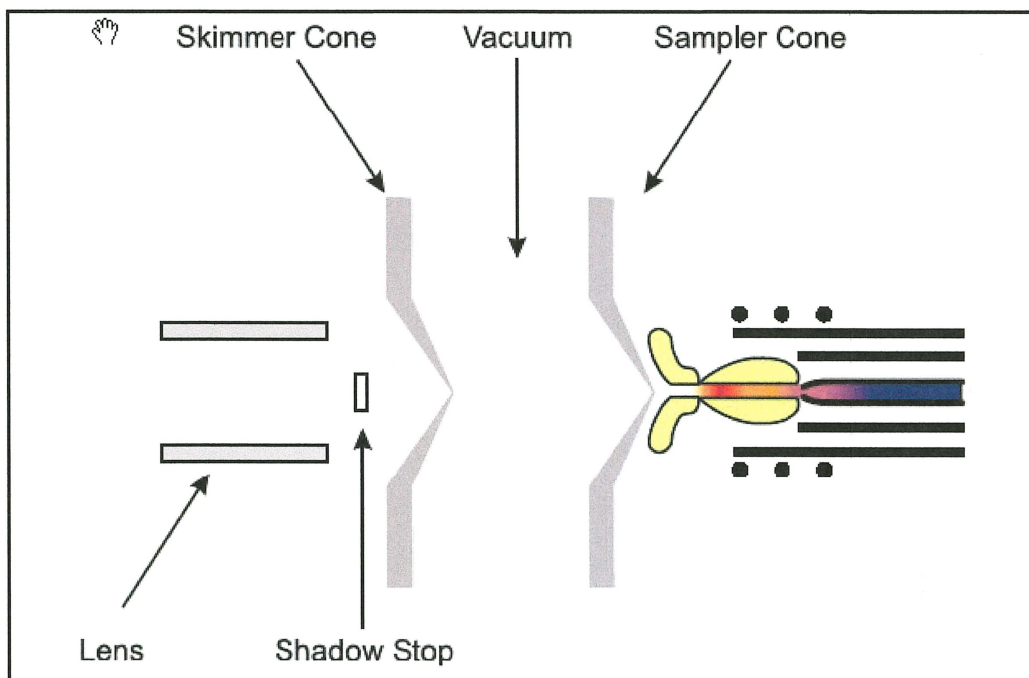


Figure 10 : Interface Region of ICP-MS

ICP-MS has many advantages over other elemental analysis techniques such as atomic absorption and optical emission spectrometry, including ICP Atomic Emission Spectroscopy (ICP-AES), including: Detection limits for most elements equal to, or better than those obtained by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS), Higher throughput than GFAAS, The ability to handle both simple and complex matrices with a minimum of matrix interferences due to the high-temperature of the ICP source, Superior detection capability to ICP-AES with the same sample throughout, and The ability to obtain isotopic information.

An ICP-MS combines a high-temperature ICP (Inductively Coupled Plasma) source with a mass spectrometer. The ICP source converts the atoms of the elements in the sample to ions. These ions are then separated and detected by the mass spectrometer. Figure 8 shows a schematic representation of an ICP source in an ICP-MS. A

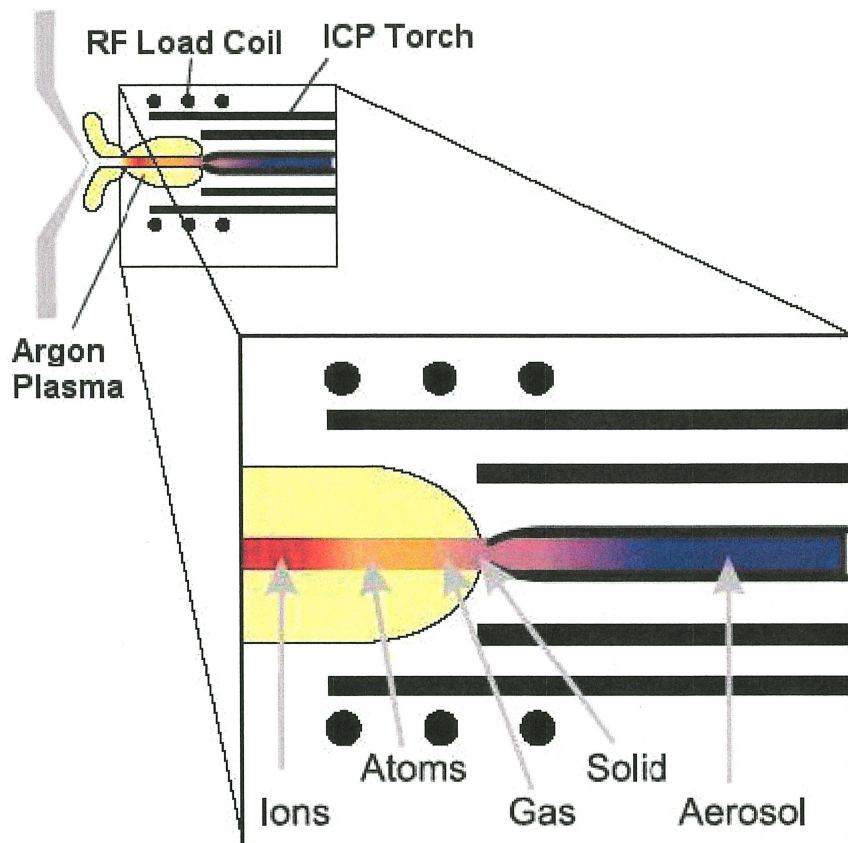


Figure 11: ICP Torch shows the fate of sample

Argon gas flows inside the concentric channels of the ICP torch. The RF load coil is connected to a radio-frequency (RF) generator. As power is supplied to the load coil from the generator, oscillating electric and magnetic fields are established at the end of the torch. When a spark is applied to the argon flowing through the ICP torch, electrons are stripped off of the argon atoms, forming argon ions. These ions are caught in the oscillating fields and collide with other argon atoms, forming an argon discharge or plasma.

The sample is typically introduced into the ICP plasma as an aerosol, either by aspirating a liquid or dissolved solid sample into a nebulizer or using a laser to directly convert solid



samples into an aerosol. Once the sample aerosol is introduced into the ICP torch, it is completely desolvated and the elements in the aerosol are converted first into gaseous atoms and then ionized towards the end of the plasma. The ions from the ICP source are then focused by the electrostatic lenses in the system. Remember, the ions coming from the system are positively charged, so the electrostatic lens, which also has a positive charge, serves to collimate the ion beam and focus it into the entrance aperture or slit of the mass spectrometer. Different types of ICP-MS systems have different types of lens systems. The simplest employs a single lens, while more complex systems may contain as many as 12 ion lenses. Each ion optic system is specifically designed to work with the interface and mass spectrometer design of the instrument.

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Of course, with every form of technology, there can be some issues that arise from using the ICP-MS. Inductively coupled plasma–mass spectrometry (ICP-MS) is a powerful analytical technique. But like any other analytical techniques, there are challenges involved. We recently asked ICP-MS experts what unresolved problems exist—especially with samples in complex matrices—and how ICP-MS methods or technologies can be developed to attack them. Craig Westphal, a principal investigator at Chemours Analytical, a subsidiary of DuPont, pointed back to sample handling. "It's now possible to use ICP-MS to analyze high total dissolved solids

samples directly at levels on par with ICP-OES, but carryover and subsequent raised instrument background levels mean we can't analyze percent and low parts-per-trillion levels easily on the same instrument, even with dedicated sample introduction systems," he said. Nevertheless, he feels that for most applications in his industrial R&D environment, existing instrumentation has enough detection limits. "Although I'd always want it to be faster, better, and cheaper," he added. Although the introduction of the collision–reaction cell greatly enhanced the ability to reduce isobaric interferences, interference removal with the CRC is matrix-dependent in single-quadrupole ICP-MS instruments, she noted—anything ionized that enters the CRC will affect the reproducibility and robustness of the cell reaction. the introduction of the triple-quadrupole ICP-MS system has been revolutionary in combating doubly charged, isobaric, and background noise interferences without losing sensitivity. In a triple-quadrupole ICP-MS instrument ionized matrix is removed in the first quadrupole, and isobaric and multiply charged interferences can be removed or reduced in the CRC, leaving the second quadrupole as a mass filter. New, integrated methods for sample preparation and instrumental analysis must be developed. For too long, instrument manufacturers have approached this issue as two separate problems, and have treated them as such—letting smaller, independent companies handle the sample handling issues while they focus only on the instrumentation itself

### *Procedure*

First, 100 mL of the stock solutions of Alkaline88, Fiji, Niagara, and Distilled water were placed in four 200ml beakers. The pH meter and electrode were calibrated by using buffers solutions of pH of 7 and 10 respectively. The electrode and glassware were rinsed with distilled water and dried properly with paper towel. 60 ml of each sample was measured and placed in 20

ml increments into three different 150 ml beakers. This step was repeated three times for each different brand of water. The pH of the water samples was recorded.

Next, to prepare the acidified water samples to be tested by the ICP, 9.44ml of each water sample was poured into 15ml container test tube. Then 0.28ml of 1% hydrochloric acid (HCl) and 2% nitric acid (HNO<sub>3</sub>) was added to each 9.44ml sample of water, making a total of 10ml. The volumes of both nitric acid and hydrochloric acid was obtained using the formula  $C_1V_1=C_2V_2$ . This process was done three times and repeated for each sample of water. With these new acidified samples, the inductively coupled plasma mass spectrometer was able to test the samples for its salt measures in the alkaline water samples.

### *Safety Precautions*

With every experiment, there is always safety precautions to prevent accidents and injuries during the lab. For handling the alkaline water, even though is water, still wear gloves, gloves and a lab coat to protect yourself from spills. For handling the ICP-MS, visitors and analysts must change to a clean lab coat designated for use inside the ICP-MS laboratories and walk through an entryway mat (sticky mat), which is set at the entering path in the sample preparation lab. Lab coat should be worn all the time in the laboratories, and cannot be taken outside the laboratories and brought back in. Gloves should be worn all the time in sample preparation lab when handling chemicals and samples. Safety goggles should be worn all the time in the sample preparation lab. Safety goggles should also be worn in the instrument lab when performing sample introduction to the ICP-MS and other instruments. All standard solutions and samples should be prepared in the Fume Hood to ensure no contaminations of the samples and all bottles containing standards or certified reference materials should be opened in

the Trace Metal Work Station. Labware and Glass wear used for solution and sample preparation should be air dried. Shorts and open-toed shoes are prohibited in the laboratories. Eating, drinking, chewing gum, applying cosmetics, taking medicine or storing food are not allowed in the laboratories.

## CHAPTER III

### RESULTS

The pH was collected from the pH node. After the over course of three trials, the average of the pHs to find the mean. Overall, the different brands of commercialized water fall around the same ph. There was no jump in data as well I the pHs. As for the different concentrations for the different salts in the water, the values of the samples were collected for Sodium, Magnesium, Potassium and Calcium. After the course of three trials, the data was averaged to find the mean of the values. As well as the mean, the standard deviation was also calculated to determine the relativity. The standard deviations for the salts show that the data is relatively close together. The mean also shows that the average value is relatively close to the values obtained from the experiment.

Table 1: pH readings for the types of water

| Type of Water   | Trial 1 pH | Trial 2 pH | Trial 3 pH | Average pH |
|-----------------|------------|------------|------------|------------|
| Niagara         | 5.58       | 5.35       | 5.35       | 5.43       |
| Fiji            | 7.11       | 6.86       | 7.10       | 7.02       |
| Alkaline 88     | 8.25       | 8.31       | 8.22       | 8.26       |
| Distilled Water | 6.72       | 6.71       | 6.76       | 6.73       |

Preparing the 2% HNO<sub>3</sub>

$$75\% \times (\text{Volume of HNO}_3) = 2\% \times 10 \text{ mL} \\ = .267 \text{ mL}$$

Preparing the 1% HCl

$$35\% \times (\text{Volume of HNO}_3) = 1\% \times 10 \text{ mL} \\ = .286 \text{ mL}$$

$$\text{Mean} = (1/n) * (N_1 + N_2 + N_3 + \dots)$$

$$(1/3) * (5.58 + 5.35 + 5.35) = 5.43$$

$$(1/3) * (7.11 + 6.86 + 7.10) = 7.02$$

$$(1/3) * (8.25 + 8.31 + 8.22) = 8.26$$

$$(1/3) * (6.72 + 6.71 + 6.76) = 6.73$$

Table 2: Salt Concentrations Results for Water Samples

| Type of Water | Type of Mineral | Sample 1 conc. | Sample 2 conc. | Sample 3 conc. | Average     | Standard Deviation |
|---------------|-----------------|----------------|----------------|----------------|-------------|--------------------|
| Niagara       | Sodium          | 0.9196 ppm     | 0.9395 ppm     | 0.9637 ppm     | 0.9409 ppm  | 0.0221 ppm         |
| Niagara       | Magnesium       | 0.0134 ppm     | 0.0109 ppm     | 0.0109 ppm     | 0.0117 ppm  | 0.0014 ppm         |
| Niagara       | Potassium       | 0.0551 ppm     | 0.0501 ppm     | 0.0521 ppm     | 0.0524 ppm  | 0.0025 ppm         |
| Niagara       | Calcium         | 0.0713 ppm     | 0.0348 ppm     | 0.0292 ppm     | 0.0451 ppm  | 0.0229 ppm         |
| Alkaline 88   | Sodium          | 1.6576 ppm     | 1.6113 ppm     | 1.7157 ppm     | 1.6615 ppm  | 0.0523 ppm         |
| Alkaline 88   | Magnesium       | 0.0197 ppm     | 0.0174 ppm     | 0.0179 ppm     | 0.0183 ppm  | 0.0012 ppm         |
| Alkaline 88   | Potassium       | 0.0875 ppm     | 0.0828 ppm     | 0.0865 ppm     | 0.0856 ppm  | 0.0025 ppm         |
| Alkaline 88   | Calcium         | 0.1140 ppm     | 0.1070 ppm     | 0.1027 ppm     | 0.1079 ppm  | 0.0057 ppm         |
| Fiji          | Sodium          | 14.7363 ppm    | 15.0356 ppm    | 14.1562 ppm    | 14.6427 ppm | 0.4471 ppm         |
| Fiji          | Magnesium       | 11.9137 ppm    | 12.2882 ppm    | 11.6265 ppm    | 11.9428 ppm | 0.3318 ppm         |
| Fiji          | Potassium       | 4.3280 ppm     | 4.4409 ppm     | 4.1317 ppm     | 4.3002 ppm  | 0.1565 ppm         |
| Fiji          | Calcium         | 8.0082 ppm     | 7.9232 ppm     | 8.0522 ppm     | 7.9945 ppm  | 0.0656 ppm         |
| Distill       | Sodium          | 0.0443 ppm     | 0.0222 ppm     | 0.0284 ppm     | 0.0316 ppm  | 0.0114 ppm         |
| Distill       | Magnesium       | 0.0017 ppm     | -0.0014 ppm    | -0.0016 ppm    | -0.0004 ppm | 0.0019 ppm         |
| Distill       | Potassium       | 0.0359 ppm     | 0.0276 ppm     | 0.0320 ppm     | 0.0318 ppm  | 0.0042 ppm         |
| Distill       | Calcium         | 0.0003 ppm     | -0.0317 ppm    | -0.0194 ppm    | -0.0169 ppm | 0.0161 ppm         |

$$\text{Standard Deviation} = [\Sigma(xi-\bar{x})^2 / (n-1)]^{1/2}$$

$$= [((5.58 \text{ ppm} - 5.43 \text{ ppm})^2 + (5.35 \text{ ppm} - 5.43 \text{ ppm})^2 + (5.35 \text{ ppm} - 5.43 \text{ ppm})^2) / 2]^{1/2} = 0.1328 \text{ ppm}$$

$$= [((7.11 \text{ ppm} - 7.02 \text{ ppm})^2 + (6.86 \text{ ppm} - 7.02 \text{ ppm})^2 + (7.10 \text{ ppm} - 7.02 \text{ ppm})^2) / 2]^{1/2} = 0.1415 \text{ ppm}$$

$$= [((8.25 \text{ ppm} - 8.26 \text{ ppm})^2 + (8.31 \text{ ppm} - 8.26 \text{ ppm})^2 + (8.22 \text{ ppm} - 8.26 \text{ ppm})^2) / 2]^{1/2} = 0.0458 \text{ ppm}$$

$$= [((6.72 \text{ ppm} - 6.73 \text{ ppm})^2 + (6.71 \text{ ppm} - 6.73 \text{ ppm})^2 + (6.76 \text{ ppm} - 6.73 \text{ ppm})^2) / 2]^{1/2} = 0.0265 \text{ ppm}$$

$$\text{Mean} = (1/n) * (N_1 + N_2 + N_3 + \dots)$$

## CHAPTER IV

### DISCUSSIONS AND CONCLUSION

In the experiment, the mineral content of each water was observed by using the ICPMS. The results showed that the Fiji water sample had the highest amount of calcium, potassium, magnesium and sodium present. This was followed by alkaline 88, Niagara and lastly distilled water. Based on these results we can infer that the higher the mineral content in each brand of water, the higher the pH the water levels. Before conducting the experiment, it was inferred that Alkaline 88 would be the most alkaline of all the samples – the results proved this theory to be accurate. The sample of Alkaline 88 showed to have the highest level of alkalinity, with an average pH of 8.26 from the conducted 3 trials. Fiji water had an average pH of 7.02, Distilled water had 6.73 and lastly Niagara had an average pH of 5.42.

In conclusion, the purpose of this experiment was to see if the pH given from Distilled water, Niagara water, Alkaline water and Fiji water followed recommended levels and also to identify the salts in each type of water: 23 Na [He], 24 Mg [He], 39 K [He], and 40 Ca [He]. Many things were learned in doing this experiment, for instance, Niagara, being the most affordable and preferred, had a lower pH than what was put on the label and the lowest pH of all the samples. Another thing that was interesting was that when testing the 3 trials for each water, the pH had gotten lower in the second and third trials of each sample. There were good amounts of salts found using the ICP except for when testing distilled water – Measures for distilled water showed mostly N/A and negative numbers as expected. There were minimal calculations done, most of which consisted of the standard deviation, the averages of the pH levels and the



concentration of the minerals for all samples. Because two sets of data were needed to calculate the t-test, it was omitted from the calculations.

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