Impact of Treatment Temperature Decline on Stability of Advanced Alkaline Biosolids

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ABSTRACT: Biosolids must be stabilized in order to reduce odors, which have been noted as a major concern with respect to alkaline stabilization. Stabilization is designed to address potential putrefaction processes, odiferous releases and vector attraction concerns. Also, most alkaline processes are open systems in which temperature and mixing are more difficult to control, and factors such as increased pressure or bactericidal action of un-ionized ammonia are not present to aid in disinfection. The purpose of this project was to begin assessment of the long-term stability of an advanced alkaline product resulting from operating conditions established by testing previously conducted and approved by EPA's Pathogen Equivalency Committee. The conditions formerly established as optimum to achieve required pathogen destruction resulted in the ability of advanced alkaline system to operate at a lower temperature of 55ºC as opposed to the temperature of 70ºC required by the U.S. EPA 40CFR Part 503 Final Rule *Standards for the Use or Disposal of Sewage Sludge*. All previous data collected regarding the ability of the advanced alkaline product to remain stabilized over long periods of time were related to the material produced at the higher temperatures which indicated no significant decline in pH over a time of 5 years.The goal of this research is to obtain better understanding of the stabilization of biosolids over time, lower costs, reduce odor formation and to reduce vector and pathogen attraction so to comply with the current requirements.

Key words: Stabilization, Alkaline, Biosolids, Closed system, Temperature

INTRODUCTION

Biosolids must be stabilized in order to reduce odors, which are a major concern with regard to public perception of alkaline stabilization (Khurram, 2011; Frahadi *et al.,* 2011; Gomes *et al.,* 2011; Trogl and Benediktova, 2011; Gousterova *et al.,* 2011; Ekmekyapar *et al.,* 2011; Krika *et al.,* 2012; Moliterni *et al.,* 2012; Lalevic *et al.,* 2012; Kim *et al.,* 2012; Castro-Gutierrez *et al.,* 2012). The process of stabilization is designed to control: (1) potential putrefaction processes, (2) odiferous releases and (3) vector attraction concerns. Stabilization can be delineated as both short and long term. Long-term stabilization is obtained when the readily degradable organics are decomposed. Processes for obtaining long-term stabilization include biological processes, such as aerobic digestion, anaerobic digestion, composting, lagoon storage and pile storage. Short-term stabilization processes, which only inhibit the putrefication process, traditionally include alkaline stabilization and heat drying processes.

Overall, stabilization is measured using well established methods that are specific to treatment processes including volatile solids reduction (VSR) and specific oxygen uptake rates (SOUR) for biological systems, pH for alkaline systems and carbon dioxide evolution for compost (Switzenbaum, *et al.,* 1997). There is concern however related to the uniformity with which these methods are conducted, particularly the methods for biological process evaluations particularly the SOUR test and VSR (Switzenbaum et al. 2002). Generally, alkaline material is defined as stable as long as the pH is held to greater than 11.5. At present, holding pH at 12 for a specified period of time delineates short-term alkaline stabilization (Ifeadi, et.al. 1975). The lime dose required to achieve the pH is related to both degradable organics and inorganics. Furthermore, alkaline stabilization requires that the pH be maintained over 12 to ensure pathogenic inhibition, a condition that historically requires longer retention time. It has been noted with open system alkaline

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treatment that the pH drops within a short period of time after lime has been added (Paulsrud and Eikumm, 1970, Doyle, 1967, Farrell et.al., 1974, Sontheimer, 1967, Counts and Shuckrow, 1974, and WEF, 1995). It has also been noted that the drop in pH level creates a favorable environment for reactivation or growth of pathogens (Wong and Fang, 2000). However, recent studies have noted the importance of ammonia in alkaline systems with regard to disinfection capacity. Pecson and Nelson (2005) demonstrated the pH is important only as it pertains to the speciation of ammonia i.e. at higher pH levels, ammonia is in the non-charged form which permeates cell membranes to achieve disinfection. This was reiterated in Pecson et al. (2007) where they noted differences in temperature, pH, and ammonia can help explain variations in Ascaris inactivation in studies on alkaline systems. Also, alkaline systems have been noted to rapidly inactivate viruses (Wei *et al.,* 2010).

With normal alkaline-stabilized biosolids, long-term storage may yield odors and pathogen regrowth and may attract pests (vector attraction) (Laor *et al.,* 2011 and Burnham, *et al.,* 1990 and 1992). Open systems require significantly higher temperature levels, consequently higher alkali doses, e.g. 0.5 to 1 kg CaO/kg DS, than those that would be adequate in closed systems (Mendez *et al.,* 2002). They also may require a longer react-retention time to achieve adequate disinfection (Bujoczek 2001). A closed system offers a number of combinations, for example: long reaction time and very low alkali dose; higher dose and shorter react time; an optimized dose at a higher pressure. However, there is not much information in the literature regarding the stability of alkaline materials from closed or advanced systems and how these process changes impact stability. One of the most prominent disinfectants associated with a closed alkaline system is ammonia. However, ammonia also has an impact on the stability of alkaline material after treatment. Once the material is removed from a closed system, the volatilization of ammonia and organo-amine compounds increases the density and activity of the beneficial microbial population. At temperatures over 52°C, ammonia and amines volatilize as a result of the following phenomena (Snoeyink and Jenkins, 1980).

a) The vapor pressure of ammonia is increased by one order of magnitude as the temperature is increased to 52°C.

b) The pH and temperature influence the speciation of NH_3. At 25°C and pH 8.5, 10% free ammonia would be present. When the temperature is raised to 50°C and the pH is held at 8.5, 50% free ammonia would be present, but at a pH of 9.5, 90% free ammonia would be present.

Ambient CO_2 in the air will also aid in the reduction in potentially inhibitory hydroxide content in the biosolids by producing calcium carbonate (an inert material) as it is absorbed in the biosolids. The net effect is to enhance biostabilization. Previous studies have indicated a balance between lime dosage and sludge is key to maintaining pH levels while minimizing costs. In a study by Farzadkia et al. (2009), they determined the optimum ratio of hydrated lime to dry weight of sludge was 0.265. At this level, the pH remained above 12 for 50 days and no regrowth of fecal coliform was noted. However the authors also note the importance of site-specific testing to determine the optimal dosage due to inherent differences in sludge characteristics. Samaras et al. (2008) examined the use of fly-ash as an alkaline agent to be used alone or in combination with lime for the stabilization of sewage sludge. They found lime to be more effective at maintaining a pH of 12 – 13 over 35 days as compared to fly ash (pH of 7.5 – 10 over 35 days). They attributed slight variations in pH over time to the reaction of calcium ions with atmospheric carbon dioxide. They also noted variation in percent solids from 20 – 50% where lower content in the 1:2 fly-ash to sewage sludge ratio. Over the 35 day test period there was not significant variation within each sample type. In both of the above studies, open containers were used for lime/fly ash mixing.

The purpose of this project was to begin assessment of the long-term stability of an advanced alkaline product resulting from operating conditions established by testing previously conducted and approved by EPA's Pathogen Equivalency Committee. The conditions formerly established as optimum to achieve required pathogen destruction resulted in the ability of advanced alkaline system to operate at a lower temperature of 55°C as opposed to the temperature of 70°C required by the U.S. EPA 40CFR Part 503 Final Rule *Standards for the Use or Disposal of Sewage Sludge*. All previous data collected regarding the ability of the advanced alkaline product to remain stabilized over long periods of time were related to the material produced at the higher temperatures which indicated no significant decline in pH over a time of 5 years. The goal of this research is to obtain better understanding of the stabilization of biosolids over time, lower costs, reduce odor formation and to reduce vector and pathogen attraction so to comply with the current requirements.

MATERIALS & METHODS

The primary samples were taken at Glendale Wastewater Treatment facility of Lakeland, Florida on July 14, 2007 from 9:00 AM – 11:30AM. The influent

source for the advanced alkaline process was waste activated sludge. As part of the initial test run, the influent sludge characteristics were monitored along with the effluent. However, only the advanced alkaline product was held for the duration. Two runs were conducted at Lakeland – one at 55°C (LBIO-55) and the other at the regulatory mandated temperature of 70°C (HBIO-70) in order to compare the pH of both runs over time. The run times were 45 minutes for the 55°C run and 55 minutes for the 70°C run. Approximately 15 gallons of each material were collected.

The above Fig. 1 shows the field placement of the LBIO-55 and the HBIO-70. A moisture permeable bedding liner was used underneath the samples and plastic fencing surrounded the sample area to prevent birds and other animals from entering the test site. A tarp was placed over the sample area to prevent rainfall accumulation in the sample. Triplicate samples of each biosolid type were taken on a weekly basis from the site described above.

The monitoring methods of the 503 regulations were implemented to determine if the pH remains stable over time and also to identify differences between the material run at 55°C and that from the 70°C run. The parameters of importance that were examined include:
- pH
- pH change with storage
- Moisture
- Temperature

pH was measured by combining 10g of the alkaline material with 20 ml deionized water as recommended in Method 4500 H⁺ of Standard Methods for the Examination of Water and Wastewater (Eaton *et al.,* 2005). A Waring two speed laboratory grade blender was used to combine the samples with the DI water as necessary. Following calibration, the pH of the slurry was then measured using an Accumet AB15 BioBasic pH meter (accuracy to 0.01). Temperatures were maintained at ambient (25°C) therefore a correction factor was not required.

Temperature and humidity were monitored on site and verified using National Weather Service data for the Statesboro, Georgia area.

Solids were tested using Method 2540 G of Standard Methods for the Examination of Water and Wastewater (Eaton *et al.,* 2005). Analysis of the data was performed using SAS, where the tests performed were non-parametric test, regression, test of location, and scale differences. Single imputation was used in SAS to input missing data, where a value was substituted for each missing value.

RESULTS & DISCUSSION

In comparing the pH levels from the advanced alkaline product from 55°C (LBIO-55) run and the 70°C (HBIO-70) run, it was found that on average HBIO-70 has a higher pH level than LBIO-55. There is a significant difference in the means of the two sample types. Though, the pH level of the LBIO-55 dropped faster, at about the 12th week, both samples remained above 12 for approximately 3 months. The HBIO-70 remained above 12 until between the 14th and 15th week and spiked above 12 in weeks 22 and 24.

One interesting point is that when one overlays the temperature readings (Fig. 2), the pH levels dropped at about the same time the average temperature dropped from 76°F to 59°F. The average temperature in the first 12 weeks was 80°F followed by an average of 60°F for weeks 13-36. In doing correlation analysis, there is an 83% positive correlation between the average pH level for the LBIO-55 and the average daily temperature. It was not as high for the HBIO-70, the correlation was only 60% between the two factors. There was very little positive correlation between the pH level for LBIO-55 or HBIO-70.

Towards the end of this experiment, the pH level of LBIO-55 seemed to stabilize more than that of the HBIO-70 biosolids. The pH level averaged about 8.5 after the 21ˢᵗ week and there was very little variability. Fig. 2 shows that the temperature stays fairly consistent
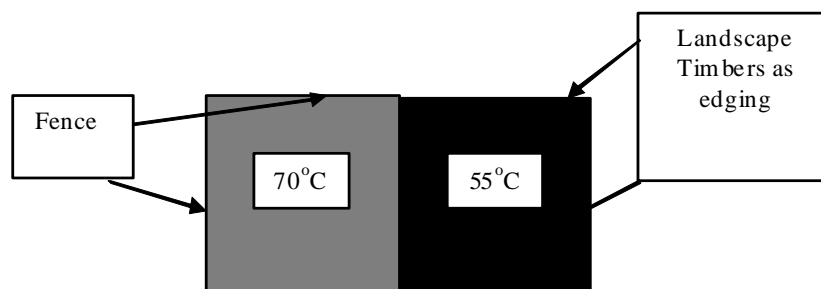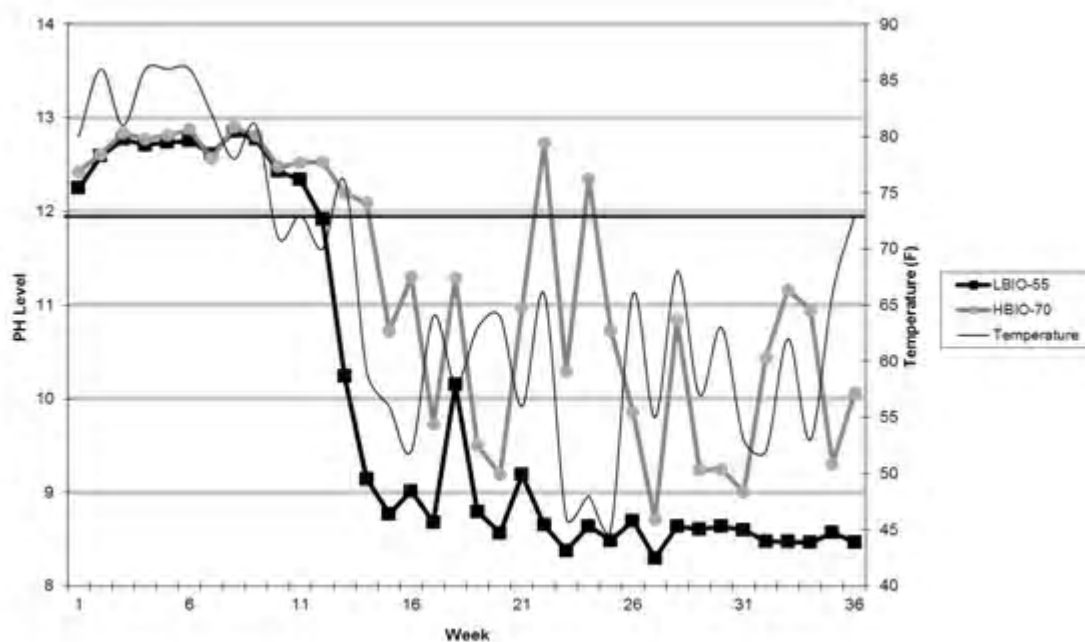

Fig. 1. Schematic of Sample Site

Fig. 2. Correlation of pH and Ambient Temperature
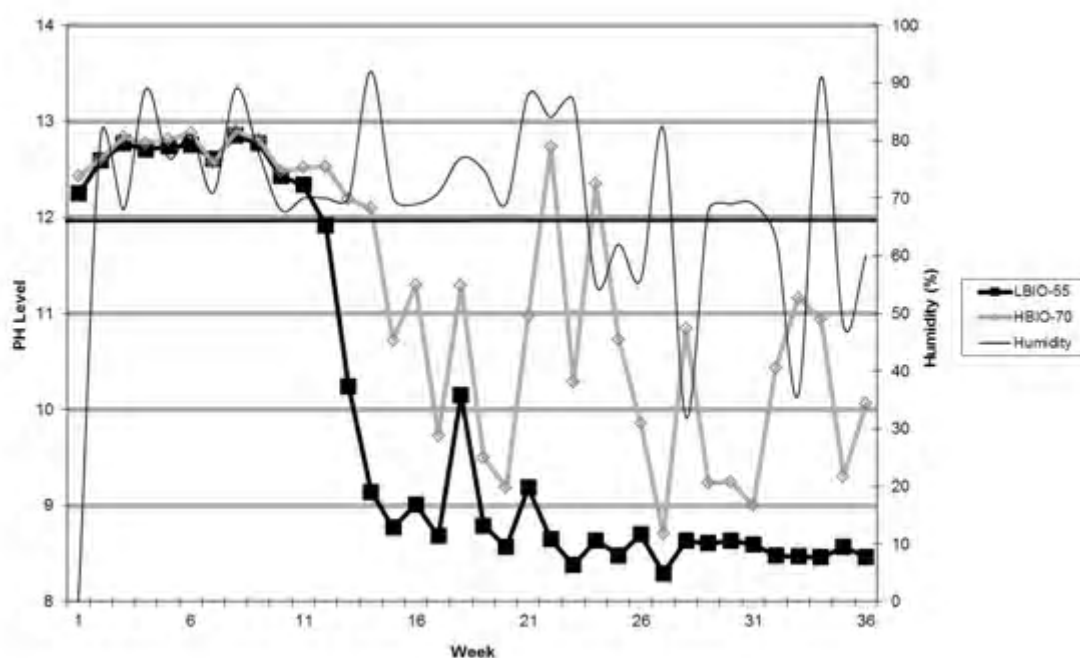

Fig. 3. Correlation between pH and Humidity

with the LBIO-55 and the HBIO-70. The important point to note with regard to the pH leveling at 8.5 in the LBIO-55 is the control by HCO_3^-. The pKa of the H_2CO_3/HCO_3^- equilibrium is 6.4 and the pKa of the HCO_3^-/CO_3 equilibrium is 10.4 resulting in 98% HCO_3^- at pH 8.5 Fig. 3 shows that there is a weak correlation between humidity, LBIO-55 and HBIO-70 pH levels. As humidity increases around Week 12, the pH level of both LBIO-

55 and HBIO-70 decrease, showing that humidity did not have a significant impact. The increases and decreases in humidity do not follow the same changes in pH levels for LBIO-55 or HBIO-70.

Fig. 4 shows that as the LBIO-55 pH decreases (Week 12), the percent solids increase. There exists a weak correlation between percent solids and pH with LBIO-55, for example, at Week 28 the pH begins to

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stabilize, but the percent solids continues to change. The correlation for percent solids and pH level was about -79% for LBIO-55.

Fig. 5 shows a more constant % solid than with the LBIO-55. In Week 12, the % solid begins to become more stable throughout the remaining experiment. The pH begins stable and fairly constant, but then begins to decrease around Week 9 until Week 14, then becoming more variable. This graph also shows a weak correlation (-63%) between pH and % solid as the changes do not follow one another each week.

The non-parametric graph shown in Fig. 6 exhibits the difference between the means of LBIO-55 and HBIO-70. The two means are not significantly different at the beginning of the experiment, but gradually the difference in the means increases over time. Regression data/graph also shows that slopes were not significantly different, with LBIO-55 having a slope of -.12 and HBIO-70 having a slope of -.10. This shows that there is no significant difference in rate of decrease for the LBIO-55 and the HBIO-70. This data is important in comparison of the two sample types. With this data,
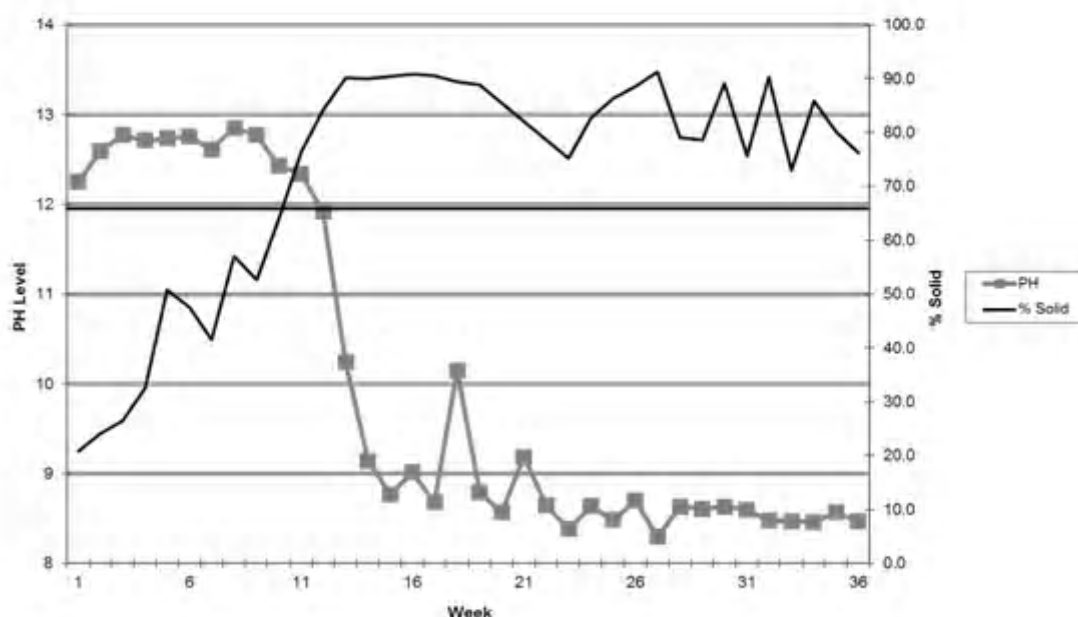

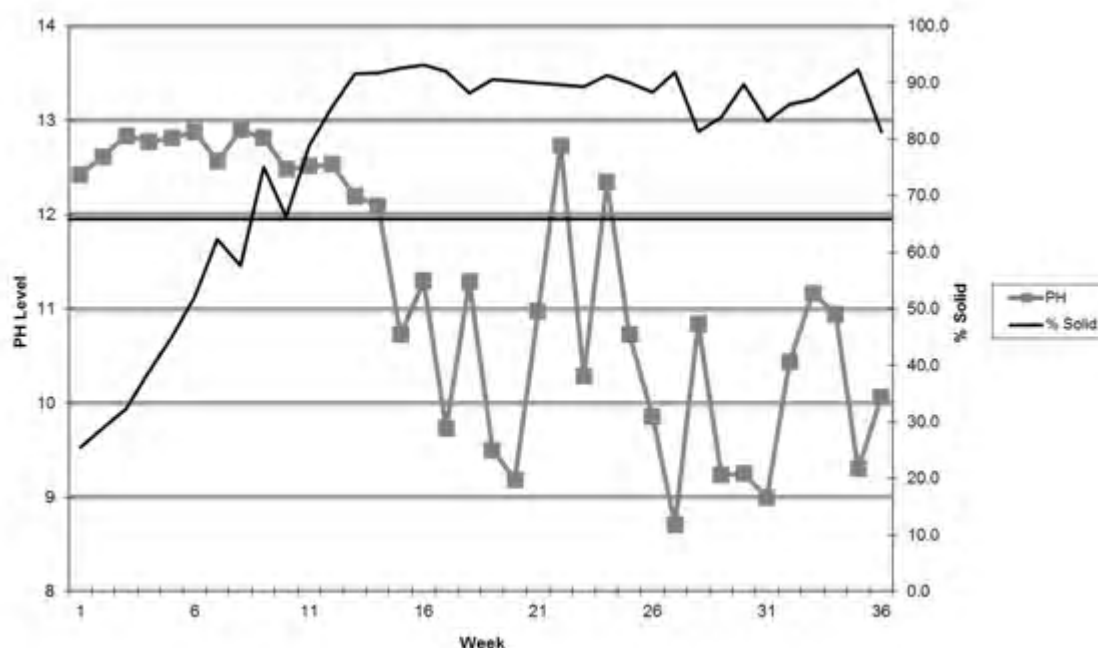**Fig. 4. Correlation between Percent Solids and pH of LBIO-55**

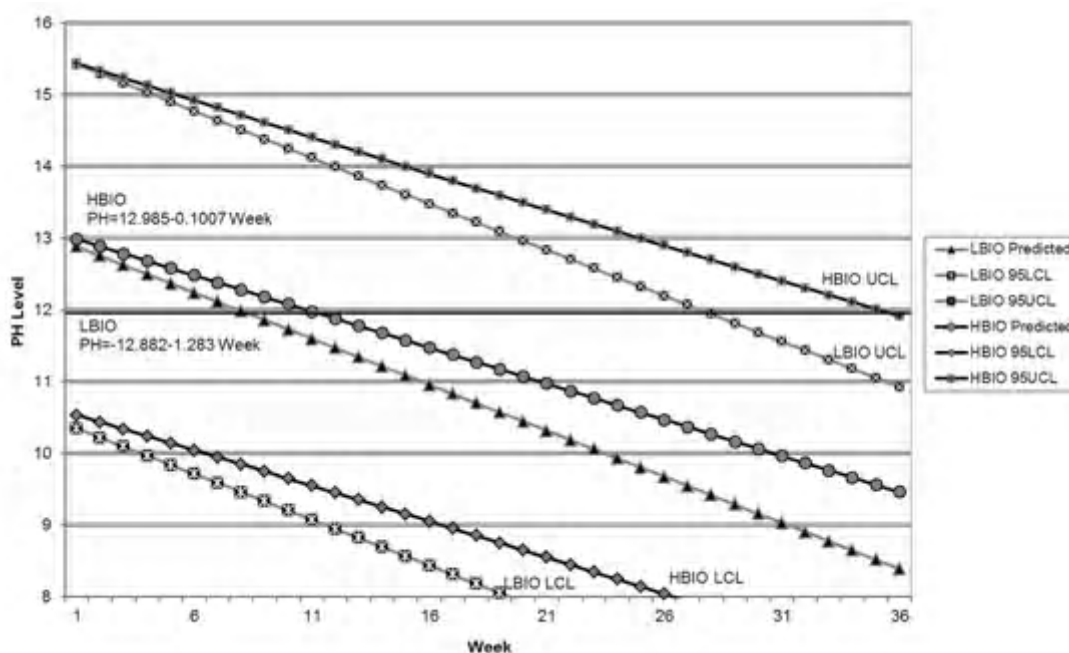**Fig. 5. Correlation between Percent Solids and pH of LBIO-70**

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Fig. 6. Estimated Regression for LBIO-55 & HBIO-70

it can be inferred the pH of the material run at the lower temperature and with less lime (LBIO-55) does not decrease faster than the pH of the sample run at the higher temperature at with more lime (HBIO-70).

CONCLUSION

The most important factors discovered with regard to the examination of the long-term stability of the representative advanced alkaline system are:

- The pH of both sample types held above pH 12 for approximately 3 months.
- There is a significant difference between the means of the pH of the samples run at 55°C and 70°C
- There is not a significant difference between their rates of pH decrease

The differences are not significant between the rates of pH decrease for LBIO-55 and the HBIO-70, as indicated by the regression and non-parametric examination of the data. However, the means of the two sample types show that there is a significant difference as the time of the experiment progressed. It is also worth noting that in Week 12-13, the temperature decreased in parallel with the pH decrease of LBIO-55. This suggests a relationship between temperature and pH, however the experiment would need to be repeated under different temperature conditions to determine the exact relationship.

To solidify the temperature / pH relationship, further testing needs to be conducted to examine the impacts of various seasonal conditions. Future work is needed to further delineate the characteristics such as oxygen uptake rates that can impact long-term stability. The SOUR (specific oxygen uptake rate) test measures of the rate of oxygen utilization in a wastewater mixed liquor or sludge. These rates can be measured using a respirometer which is a set-up that allows the system of various sludges to be monitored over a longer period of time and also provides direct output of the oxygen uptake curve. Oxygen uptake rates are able to delineate the effectiveness of ammonia disinfection and neutralization by measuring the amount of microbial activity within the treated biosolids.

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