SOME PHYSICAL CHARACTERISTICS AND HEAVY METAL ANALYSES OF COTTON GIN WASTE FOR POTENTIAL USE AS AN ALTERNATIVE FUEL

THESIS

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By

Sungsoo Kim, B.S., M.S.
Denton, Texas
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Utilizing cotton gin waste as a fuel is an attractive solution to the problems of disposing of a surplus agricultural waste as well as supplementing energy resources. Because a qualified alternative fuel must meet both environmental emission standards and industrial fuel standards, the physical and chemical characteristics of cotton gin waste and its toxic element concentrations are important for its objective evaluation as a fuel.

Constituent components, moisture contents, and ash contents of four separate parts of cotton gin waste were determined and evaluated closely following the American Society for Testing and Materials (ASTM) test methods. The three most toxic heavy metals, Arsenic (As), Chromium (Cr), and Lead (Pb), chosen for quantitative analysis were determined by using an inductively coupled plasma atomic emission spectrometry and a microwave oven sample digestion method.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF ILLUSTRATIONS

CHAPTER I

INTRODUCTION

Cotton Gin Waste

During the four year period 1991 through 1994, an average of 1 million tons or 4.4 million bales per year of cotton was produced in Texas (1). All of the cotton produced in Texas today is harvested by machine. There are two types of machine harvesters: **(1)** spindle pickers and **(2)** strippers. Pickers are used primarily in areas where the growing season is long, the cotton matures over an extended period of time, and more than one picking is required. They are most commonly used in the irrigated areas of South and Southcentral Texas. Since pickers are designed to be a more selective method of machine harvesting than strippers, the amount of waste that is collected in the harvesting process is considerably less.

Cotton strippers go over the field only once, after the plant is desiccated either by frost or by the application of chemicals. In the process of harvesting, strippers collect a much larger quantity of leaves, burs, stalks, other plant materials, and soil particles than do pickers. Strippers

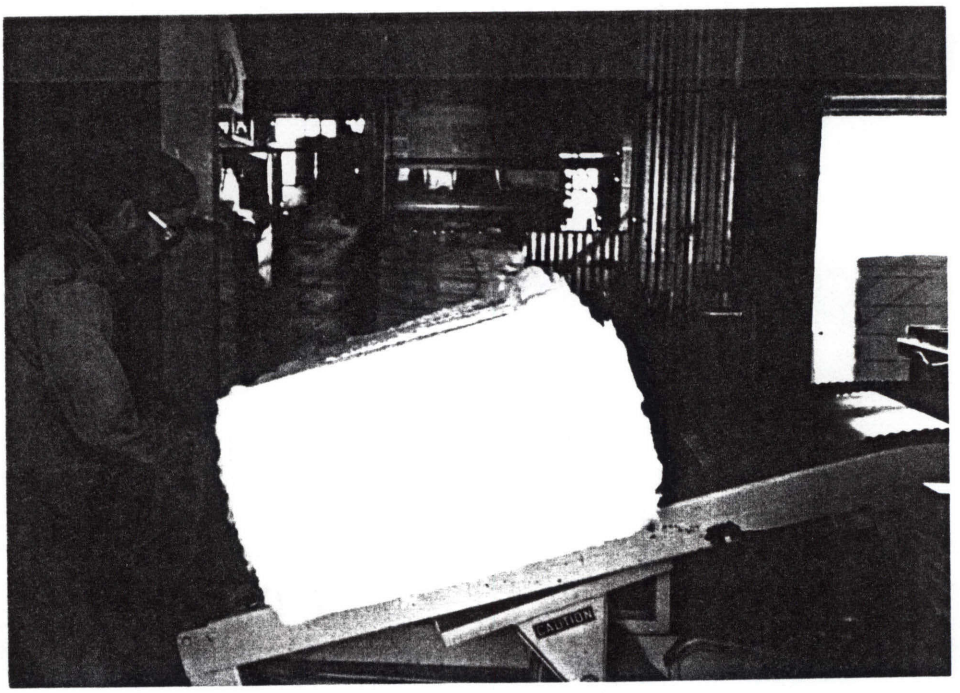
harvest about 70 percent of all cotton in Texas (2). The cotton harvesting strippers primarily used in Texas are shown in Figure 1.

All material collected by the harvesting machines is transported to gins, which separate lint, seed and foreign matter. The amount of foreign material of gin waste that is collected varies considerably according to season, geographic location and the harvesting process. Previous research indicates that it averages close to 50 kg per bale of spindle-machine-picked cotton and 300 kg per bale for stripper-harvested cotton (3-5). Table 1 shows some of the values reported by investigators for the amount of waste material produced from the seed cotton which is required to

TABLE 1. ESTIMATED AMOUNT OF COTTON GIN WASTE PER BALE (218 Kg lint) OF COTTON

	Harvesting Method	
Investigators	Mechanically stripped	Mechanically picked
Lalor and Smith	294 Kg	60 Kg
Pendleton and Moore	238 Kg	37 Kg
Alberson and Hurst	270 Kg - 455 Kg	54 Kg - 68 Kg

FIGURE 1. COTTON HARVESTING STRIPPER



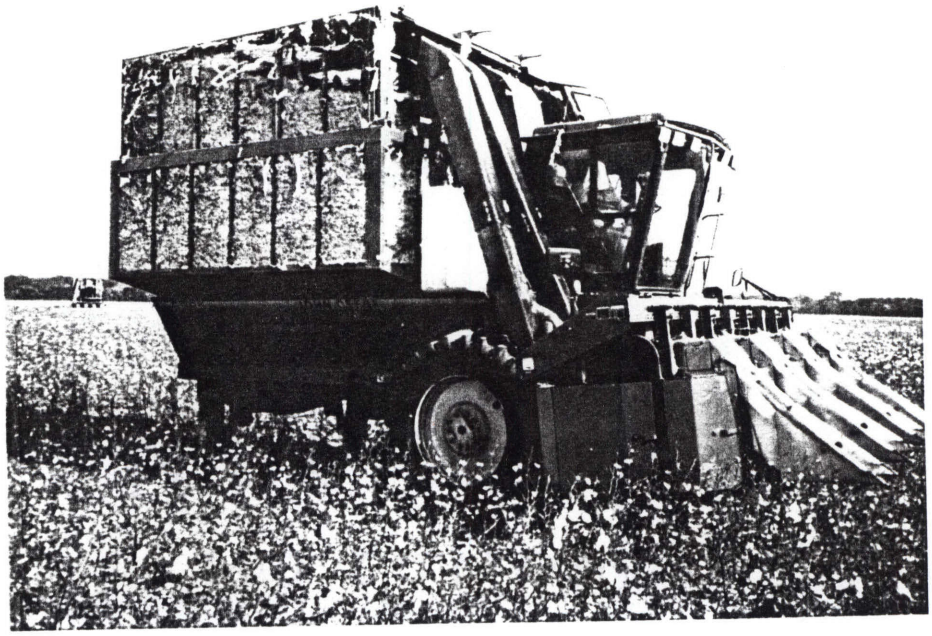
produce a 218 kg bale of lint cotton. Figure 2 shows the picture of the 218 Kg bale of pure lint cotton. It reflects that approximately 1 million tons of cotton gin waste are produced in Texas alone.

Disposal Methods of Cotton Gin Waste

Even though the number of active gins in the United States has been decreasing during the past decade, the productivity of cotton per gin factory has been increasing. Hence the amount of waste that accumulates at gins is becoming much more massive and has constituted a more severe disposal problem for many gins than in previous gins. Until prohibited by atmospheric pollution regulations, gin operators usually disposed of the waste by open burning. The most prevalent disposal method currently used is the direct return of the cotton gin waste to the land. This method is not practiced in cases where certain diseases or weed problems exist and their spreading is undesirable.

A secondary method of disposal is the feeding of cotton gin waste as roughage in livestock rations. As the gin operators were forced to find new outlets for the previously burned waste, the cattle feeder found an easily procured product. Nevertheless, addition of cotton gin waste to rations has not found widespread acceptance in the livestock

FIGURE 2. 218 Kg BALE OF PURE LINT COTTON



feeding industry. This is mainly due to its high chemical residues such as pesticides and harvest aid chemicals, etc.

Reddell et al. (6), and Alberson and Hurst (4) studied composting as a means of **(1)** reducing the volume of cotton gin waste to be eventually disposed of and **(2)** elimination of the potential of weed and disease spreading when gin waste is applied back to the land. However, composting of cotton gin waste has not been accepted widely as a means of waste disposal, apparently because of the increased cost of handling and the increased fire hazard. Another obstacle of composting is that large flat land areas are required in order to facilitate composting the volume of material left from even a small gin.

Previous Studies of Cotton Gin Waste as an Energy Source

The large quantities of cotton gin waste available, coupled with the high demand for new alternative energy sources, has brought increasing attention to the potential of cotton gin waste as a source of energy. Approximately 4 to 5 million bales of cotton are ginned in Texas annually, most of which are stripper-harvested. Approximately 300 kg of gin waste are associated with each bale of stripper-harvested cotton, and each pound of gin waste can yield

about 7,000 Btus. Thus the potential energy resource appears large enough to warrant this research.

Griffin (7) first reported the heat value and ash content of cotton gin waste. The determinations were done on waste from mid-South picker-harvested cotton during the 1973 ginning season. Heat value of the gin waste was about 7,000 Btus per a pound of dry waste, and the ash content was about 10 percent.

The formation of rounded, glass-like particles in the firing cup was also detected by Griffin (7), and he attributed these to the sand in the material tested. Glass buildup on the walls and floors of an incinerator under high temperature incineration of cotton gin waste was mentioned as a possible problem.

Research conducted by McCaskill and Wesley (8) was focused towards utilization of heat given off by burning gin waste for drying seed cotton during the ginning process. The primary objective was the development of an acceptable waste incineration disposal system for the ginner. There are no incinerators specifically approved for cotton gin waste disposal, so this study was a logical step in the direction of the development of a heat-recovering incinerator system.

The incinerator used in this study utilized machine-picked cotton waste as fuel. Machine-picked cotton normally has less than one-sixth the waste of machine-stripped cotton. Even with this relatively small quantity of waste, the research showed that with the achieved 30 percent overall recovery, the system could supply the heat required for drying the seed cotton to an acceptable level. The opacity of the stack discharge has been estimated as less than 10 percent.

LePori et al. (9) included an example to demonstrate the feasibility of using agricultural residues for energy. A cotton gin that is ginning stripper harvested seed cotton produces enough energy, in the form of cotton gin waste, to power the gin even if the overall conversion efficiency is as low as 3.5 percent. Any unused heat from power generation could be used to dry the seed cotton if necessary.

Beyond the incineration of cotton gin waste for energy production, there are other possibilities. Benedict and his associates (10) investigated the possibility of hydrogen gas production and protein extraction from cotton gin waste through an enzymatic process. Only the water soluble portion of the waste is utilized and the fibrous material remains.

The conversion of agricultural residues to char, condensable gases, and non-condensable gases using pyrolysis has been studied and reported by Knight et al. (11). Laboratory bench scale and pilot plant experimental work have been accomplished using a number of materials. Materials that have been processed include pine bark, pine sawdust, cotton gin waste, hardwood chips, peanut shells, nonmetallic automobile waste, and municipal waste. One distinct advantage of fuels produced from the agricultural materials is the low sulfur content. Air emissions from facilities using these fuels would then be very low in sulfur dioxide. A possibility was described of mixing low sulfur pyrolitic char and condensable gases with high sulfur coal in a ratio such that the mixed fuel is within the allowable sulfur limits for solid fuels.

From these short and brief reviews, it can be easily realized that the utilization of the waste for energy production is still at an entry level. It has been hampered by many problems involved with using the waste for energy, such as the difficulty of handling gin waste, the high content of fine dust in gin waste, the uncertainty of being able to establish reliable market outlets for the energy produced, and certain environmental questions.

Toxic Metals in Cotton Gin Waste

Surprisingly, despite the fact that cotton gin waste has a high content of environmentally hazardous pesticides or harvest aid chemicals which contain some toxic elements, very little work has been reported on heavy metal content of cotton gin waste. Only arsenic residue in cotton gin waste has been of some concern (12-14). Arsenic acid (as a principal chemical desiccant) has been actively used to prepare cotton for harvesting by stripping, for the past four decades, especially in Texas and Oklahoma. However, arsenic acid is no longer used as an approved harvest aid chemical in Texas and Oklahoma. It was last used during the 1993/94 cotton harvest (15). Therefore, it is assumed that all future gin waste will not contain any arsenic from the harvesting process.

Nonetheless it would be worth investigating how much arsenic residue still exists in cotton gin waste due to the soil contamination of cotton fields with regard to the long term use of arsenic acid. Hence, this study is mainly focused on arsenic content in the waste left from the cotton harvest of the 1994/95 season, which is the first without arsenic acid.

Besides arsenic, many other toxic elements can also exist in cotton gin waste. For reference, a semi-quantitative

analysis of 13 environmentally toxic elements in the waste was conducted by Southern Spectrographic Laboratory in Irving, Texas, under contact from my major professor, using Inductively Coupled Plasma Atomic Emission Spectrometry. The results are included in Table 2. Based on these results and the extent of toxicity, Arsenic (As) and two other toxic heavy metals, Chromium (Cr) and Lead (Pb), which showed high concentrations were chosen for a full scale quantitative analysis.

For reference, a summary of toxic effects of the three heavy metals, which were analyzed in this research are listed.

ARSENIC

Exposure to or poisoning by arsenic can result in diarrhea, severe colic, bloody feces, reproductive system problems, cirrhosis of the liver, nervous disorders, kidney disorders, and skin disturbances (16-20).

CHROMIUM

Chromium is one of the elements that exhibit a dual nature from a toxicological and an essential element standpoint. It is essential for the normal metabolism of glucose. Chromium compounds are suspected carcinogens, as evidenced by the cancer rate in the chromate-producing

TABLE 2. SEMI-QUANTITATIVE ELEMENT ANALYSIS OF COTTON GIN WASTE

ELEMENT	CONCENTRATION*
As	5 PPM
Cd	not detected
Hg	not detected
Pb	30 PPM
Sb	not detected
Se	not detected
Zn	40 PPM
Tl	not detected
Ba	8 PPM
Be	48 PPM
Cr	28 PPM
Co	23 PPM
Ni	29 PPM

*: Some of the elements (Pb, Zn, Be, Cr, Co, and Ni) have distinctly high concentrations when compared to the others. It was strongly suspected that the sample was contaminated from a crucible during grinding or pulverizing of the sample. Therefore, grinding procedure was eliminated during sample preparation for quantitative analysis.

industry. The rate is 28 times that of the general population. Chromium causes perforation of the nasal septum, congestion, hyperemia, emphysema, tracheitis, pharyngitis, broncho-pneumonia, dermatitis, and metal fume fever (16-18, 20, 21).

LEAD

Toxic effects of lead are well documented. Lead tends to accumulate in the body; it is excreted to only a slight degree. Lead is deposited in the bone and the soft tissues--particularly the brain, where it results in reduced functioning. Lead can cause structural damage to mitochondria of the kidneys resulting in the loss of amino acid, glucose, and phosphate in the urine. Lead has also been linked to increased dental caries, intestinal colic, peripheral neuropathy, and encephalopathy. Symptoms of lead poisoning include headache, fatigue, and weight loss (16-18, 22-24).

Summary

Cotton Gin Waste (CGW), one of the diverse agricultural wastes, is a potential alternative energy source to the otherwise rapidly depleting fossil fuels. Approximately 1 million tons of cotton gin waste, out of a total 1.7 million tons produced in the whole United States, are produced

annually in Texas (1). Despite the strict enforcement of environmental regulations, there is only very little progress in the development of alternative methods, e.g., composting, fuel, and cattlefeed, to traditional methods such as incineration or landfill for the effective utilization and safe treatment of cotton gin waste. Nowadays cotton gin waste is generally being distributed as raw original gin waste in the fields because of EPA or state regulation limiting landfill and open burning of the waste. Even though various possible methods of conversion of cotton gin wastes to energy sources have been widely studied since the mid-1970s, they have not been successful. This has been mostly due to two limiting factors.

First, modern production of cotton is accomplished by the substantial use of chemicals like pesticides and harvest aid chemicals, etc. The categories of special interest among those chemicals are the harvest aid chemicals such as defoliants and desiccants listed in Table 3, mainly because of their use just before the cotton harvest. It has long been recognized that this chemical usage leads to relatively high levels of residues in cotton gin waste (25-27). Surveys in Texas and California (25, 27) have shown that cotton gin waste almost always contains residues ranging between 10 and 225 PPM (μg/ml) of those chemicals. Among

those chemical residues, the content of arsenic residue in particular, was exceptionally high in cotton gin waste. Notwithstanding the fact that the use of arsenic acid as a desiccant was banned from the 1994/95 season of cotton harvest, it is highly suspected that the gin waste still contains a significant amount of arsenic. Thus it should be carefully considered that the residual chemicals, especially arsenic residue, in cotton gin waste, may create a problem during its combustion as a clean fuel.

Second, cotton gin waste consists of burs, bits of lint, sticks, and fine dust. The fine dust, commonly referred to as "fines," makes up to 33% by weight and 20% by volume of cotton gin waste (27). Presence of high content of fine dust in the waste constitutes one of the problems in its utilization as a source of energy as it will leave a lot of ash during the combustion process. For the successful utilization of cotton gin waste as an energy source, fine dust must be removed before its use as a fuel and an effective fine dust removal method needs to be developed.

Nonetheless, cotton gin waste is a potential energy source, mainly because of its reasonably good heat content of about 7,000 Btus per pound of material (7) and its availability. Low emission of sulfur and low ash content from the combustion of pure cellulosic parts of cotton gin

TABLE 3. AVERAGE HARVEST AID CHEMICAL CONTENTS OF COTTON GIN WASTE IN TEXAS

Harvest aid chemicals	Cotent (PPM)*
Defoliants	
DEF(S,S,S-tributylphosphorotrithioate)	7.0
Folex(S,S,S-tributylphosphorotrithioite)	9.0
Cacodylic acid(Arsenical)	18.0
Desiccants	
Arsenic acid**	225.0
Paraquat(Chlorinated)	9.5

*: PPM = μg/ml

**: Currently not used.

waste (11, 28) suggest the possibility of the waste as a clean fuel. In addition, a promising new market that can utilize agricultural wastes as pellet fuel is quickly growing in the United States (29). Accordingly, the commercialization of pelletized agricultural waste is being watched with considerable interest. Consequently, solving the above mentioned problems will decide whether or not cotton gin waste can become an attractive and competitive energy source.

An essential key for the successful conversion of cotton gin waste into an alternative fuel is that emissions from combusted cotton gin waste must meet environmental emission

standards. Several fundamental physical characteristics of cotton gin waste, which are other important factors for the successful utilization of cotton gin waste as an energy source, must also meet minimum industrial standards as a fuel (30). Hence, as a part of these needs, this research has carried out the investigation of some important physical characteristics and toxic element analysis of cotton gin waste.

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CHAPTER II

METHODOLOGY

This chapter is divided into two parts. The first part describes the determination of physical characteristics of cotton gin waste, and the second part includes the instrumentation of Inductively Coupled Plasma (ICP) and the elemental analysis of ICP.

PHYSICAL CHARACTERISTICS OF COTTON GIN WASTE

Constituent Parts

Cotton gin waste, collected at Modern Gin in Buckholts, Texas in August of 1995, was physically separated into four different parts--i.e., burs, sticks, lint, and fine dust--using ten different sizes of ASTM standard testing sieves continuously stackable. The size of those sieves varies from 5 mm to 38 μm. Approximately 8 g to 49 g of composite waste were randomly picked by hand eleven times. After initial mass determination of each sample, the long-term task of separating the quantity of waste into four separate constituents was begun. Each of the 11 specimens was placed on the top of the sieves stacked continuously and shaken for

30 minutes using a testing sieve shaker from Ro-Tap®, Model B, Combustion Engineering Co., Inc., Houston, Texas. After being roughly separated, dissection microscope and magnifier were used to separate very small foreign matter from the separated lint, burs, and sticks. The material that fell through the 4 mm sieve was referred to as fine dust. Any small pieces of lint, burs, and sticks that fell through the 4 mm testing sieve were removed from fine dust each to the corresponding components. Separation continued until it was not feasible to search any longer for the various components. Each portion--burs, sticks, lint, and fine dust--was placed on the balance and the mass determined. From the mass of each constituent part, the total mass and the percentage of the total due to each was determined. The difference between the beginning mass and the sum of constituent masses, except sample No.1, was calculated as percent loss. It ranges from 0 % to 3.2 %. This loss can be attributed to both the particles and the moisture being lost during handling.

Bur and stick separation was very nearly 100 percent accomplished. Some of the lint contained very small particles of leaf, stick, and other foreign matter. Even though the dissection microscope and the magnifier were used during the separation procedure, any equipment that could

remove such fine particles of foreign matter more effectively was not readily available to the investigator. Therefore, the hand separation was accomplished as best as humanly possible. Sand, soil, small sticks (less than 4 mm in length normally), and leaf particles were the major items found in the fine dust.

Moisture Content

Moisture content of the received samples was determined by the oven drying method. The mass of aluminum sample cans, 7.6 cm diameter x 2.5 cm deep with lids, was determined to the nearest 1 mg using an American Scientific Products Co., Model B1240-1, side loading balance. Approximately 0.2 g to 1.0 g each of a total 50 samples (40 specimens taken from separated cotton gin waste and 10 specimens from original cotton gin waste sample) were used for the determination of moisture content. All of the samples were tested as directed in ASTM Test Method D 2495, Moisture in Cotton by Oven-Drying. Using the balance, the combined mass of can and sample was determined.

A Modern Laboratory Equipment, Model 4-357-120, forced air drying oven was then utilized to dry the samples for 12 hours at 105 ± 4°C. After drying, desiccators were used to allow the cans and samples to cool without picking up any

moisture. Mass determinations, to the nearest 1 mg, were then made for the cans and dried waste using the same balance. Specimens and cans were returned to the oven for an additional hour of drying. The specimens were then allowed to cool in the desiccators and mass determinations made.

A difference in mass for two successive dryings of 0.1 % or greater of the most recent specimen mass called for an additional hour of drying, cooling, and mass determinations. Whenever the change in specimen mass between successive determinations was less than 0.1 %, the last mass was recorded as the final mass. Moisture content was then calculated using the equation below.

$$MC = [(M-D)/M] \times 100$$
$$M = G-E$$
$$D = B-E$$

where:
M = mass of specimen as received, g,
G = gross mass of specimen and container, g,
D = oven-dry mass of specimen, g,
B = mass of oven-dry specimen and container, g, and
E = mass of empty container, g.

The final moisture content reported for each sample is the average of the ten moisture contents calculated for the

ten specimens from four separate constituent parts of cotton gin waste. Average moisture content of original cotton gin waste was obtained from the average weight percent times the average moisture content of each of the corresponding constituent parts. For a verifying purpose, ten specimens of original cotton gin waste were examined for moisture content determination.

Ash Content

Ash content determination was made on as received test specimens. Empty porcelain crucibles were heated to 600°C, allowed to cool in a desiccator, and the mass determined to the nearest 0.1 mg, using an American Scientific Products Co., Model B1240-1, side loading balance. Approximately 0.2 g of each of the 30 specimens from three constituent parts of cotton gin waste--burs, lint, and sticks--was placed in the crucible and the total mass was determined. The recorded specimen mass was the total mass minus the mass of the crucible.

The as received specimen and crucible were placed in a Lindberg, Model 51333, muffle furnace and the temperature increased slowly up to a final temperature of 600°C. This process took approximately five hours. Crucible and contents were then removed from the muffle furnace, cooled

in a desiccator, and the mass was determined. Heating at 600°C was repeated for 30-minute periods, the crucible and contents cooled, and the mass was not determined until the mass was constant to within 0.1 mg. After the final mass determination, the ash material was placed in a 2 ml vial for later use.

Ash, percent, for the as received specimen (APAR) was calculated using the equation,

$$APAR = (W_1/W_2) \times 100$$

where:
W_1 = weight of ash, and
W_2 = weight of as received sample.

To find the ash, percent, on an oven-dried basis (APOD), the following calculation was made,

$$APOD = APAR \times \left(\frac{100}{100-MC}\right)$$

This procedure follows very closely the American Society for Testing and Materials Standard D-1102, Ash in Wood.

ICP METHOD

Historical Aspects

The development of ICPs began in 1942, when Babat published his first paper on the properties of electrodeless discharges (1). The importance of Babat's papers is that they document the first successful operation of ICPs at atmospheric pressure. The next significant development in the evolution of ICPs occurred in the early 1960s when Reed described his ingenious approach to the stabilization and isolation of these plasma. The introduction, in 1963, of the ICP as a source for analytical atomic emission spectrometry (AES) constituted a revolutionary advance in this field (2). The performance characteristics of ICP-AES, namely its versatility, wide applicability, and ease of use, are almost unparalleled among methods of elemental analysis.

In principle, any element, other than constituents of the injector gas, can be determined by ICP-AES. The accuracy, precision, and sensitivity attainable by ICP-AES are suitable for all application requirements. Furthermore, the almost invariable presence of a dedicated computer in ICP-AES instrumentation means that despite the complexities of the methods, new users find themselves rapidly producing

good results. Of all the various tools for atomic spectrochemical analysis, the ICP has had the most significant impact on the field of atomic spectroscopy.

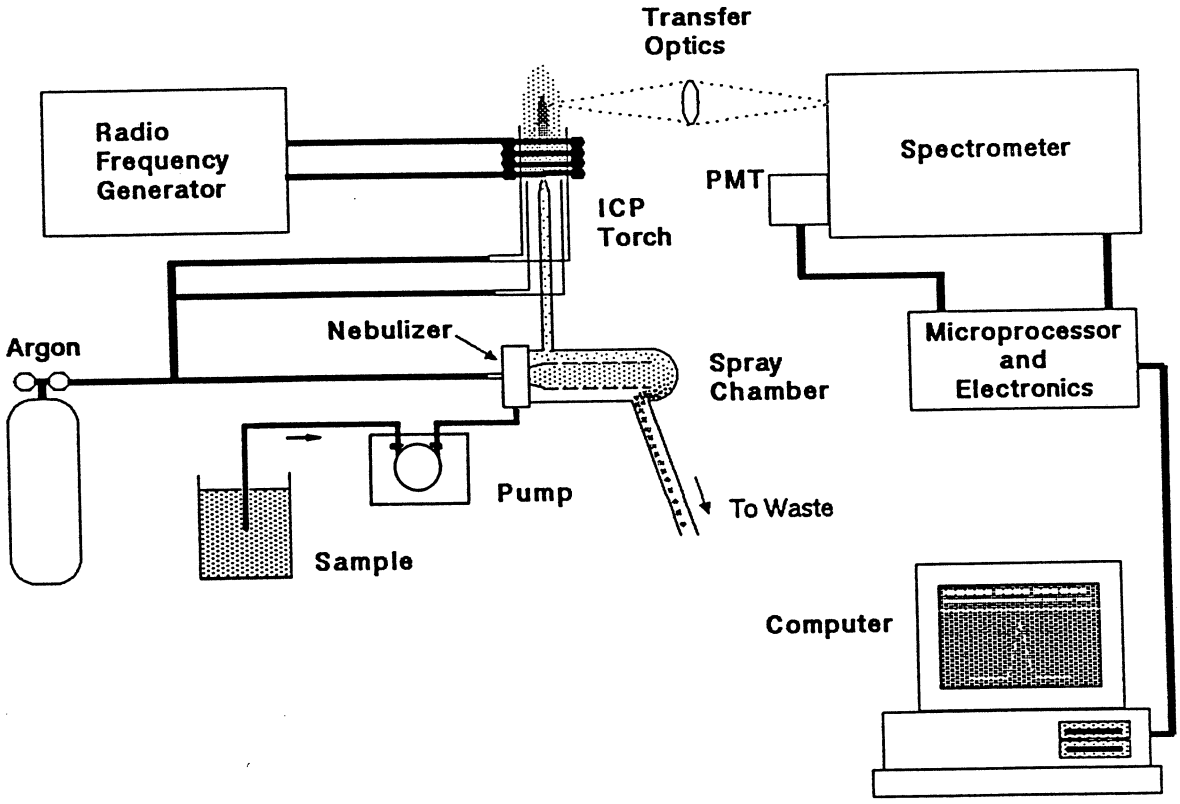
Instrumentation of ICP

The ICP instrument is comprised of five fundamental parts: radio frequency (RF) generator, ICP source, spectrometer, sample introduction, and data analysis. Major components and layout of a typical ICP-AES instrument are shown in Figure 3.

Radio Frequency (RF) Generators

The RF generators used to supply power to ICPs are oscillators that basically generate an alternating current at a desired frequency (2). The basic circuit for an oscillator consists of a capacitor and an inductor in parallel.

FIGURE 3. MAJOR COMPONENTS AND LAYOUT OF A TYPICAL ICP-AES INSTRUMENT



When the capacitor is discharged through the inductor, the subsequent collapse of the magnetic field causes charge buildup on the capacitor. The majority of instrument manufacturers use generators with nominal powers of 2 Kw and a frequency of 27.12 MHz.

ICP Sources

Historically, the ICP has comprised of a "torch," which contains the hot plasma, and some means to sustain the plasma inside the torch (3). Radio frequency power is transferred to the plasma using a coil wound around the torch. The RF coil does not come into direct contact with the plasma. This eliminates the problem of elemental contamination in the source from the electrode. This coil, once energized with RF power, induces an electromagnetic field within the torch. This field inductively heats the formed plasma to temperature exceeding 6,000 °K. The gas that sustains the plasma is initially made electrically conductive by Tesla sparks before a self-sustained plasma results.

The torch typically consists of a series of annular tubes made of quartz. The various tubes making up the torch carry gases of different flow velocities through the RF coil region. In this region, the gas is rapidly heated and

subsequently ionized. The outer stream flows at a high rate (5~20 L/min.) and serves to sustain the plasma. It also carries away the heat that is dissipated by the plasma to the inside walls of the torch. A centrally located gas stream flowing at a low rate (0.3~1.5 L/min.) carries a sample aerosol up through the existing plasma. After penetrating the hot core of the plasma, the aerosol is desolvated, dissociated, atomized, and excited. Upon passing through and out of the plasma, the various ionic and atomic species relax to their ground states, emitting characteristic radiation.

Spectrometers

The optics and spectrometer of the early ICP commercial systems were typically borrowed from established arc/spark systems concurrently available and retrofitted with the newer ICP source. Direct-reading systems capable of analyzing nearly 50 elements at one time were expensive and space-consuming. The ICP source was typically mounted on a translation table that permitted manual positioning of the ICP for optimum signal observation. Before the production and general availability of holographic grating, spectral resolution necessary for the wavelength of 0.01 nm was achieved through focal lengths of 1 m or even more. With the advent of better optical configurations using

holographic grating with 3,600 grooves/mm, more efficient methods for slewing the grating from one end of the spectrum to the other suddenly took the bulky instrument design into the more practical, bench-mountable systems. Reliability and performance of a truly sequential monochromator under computer control, not available until the late 1970s, finally stood up to customers' specifications.

Sample Introduction

Many different methods of sample introduction have been developed for the ICP. Nebulization of a liquid to form an aerosol is the most popular. A nebulizer is often used in conjunction with an aerosol discrimination chamber (or spray chamber) to form a fine mist with a mean droplet diameter of several micrometers. This mist is transported and injected into the plasma. Efficiency of these devices varies between 1% and 3% of the total solution. The remainder of the sample goes to waste.

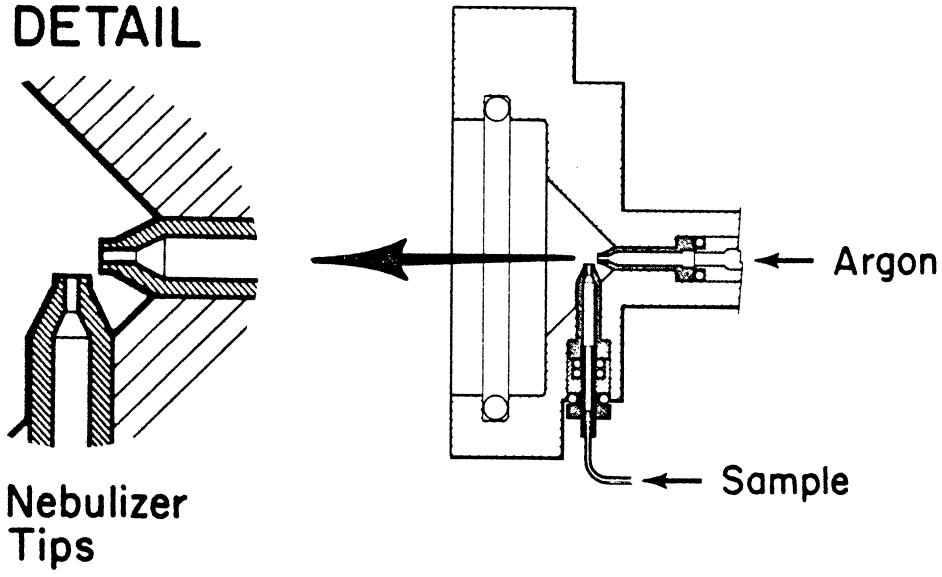
Historically, nebulizers have consisted of two glass tubes placed at right angles to one another. A horizontal tube with a 5 μm orifice transports a gas of high velocity past a vertically positioned tube through which the liquid sample flows. This "cross flow" nebulizer aspirates through a shearing action, forming droplets with a wide range of

diameters. The aerosol chamber discriminates against the larger droplets, and the smaller droplets proceed, after various further encounters with the chamber wall and other droplet particles, to the plasma. Figure 4 shows a typical "cross-flow" type nebulizer generally used in a conventional ICP analysis.

Data Analysis

Most of the developments and improvements in optical emission sequential spectrometers are directed at increasing precision of wavelength location and speed of wavelength selection by the computer. A microprocessor is often located directly inside the instrument and communicates to the user through a keyboard. The user is able to run software packages for data analysis and output. Data handling and automation are performed by the same computer used to control the process being monitored.

FIGURE 4. TYPICAL "CROSS FLOW" TYPE NEBULIZER



Detection Limits

Detection limits are important figures of merit in all branches of analytical chemistry. In routine analyses, they are intuitively interpreted as the lowest concentration that one can determine with a particular method. Detection limits are usually determined experimentally with ideal samples that contain only analytes at very low concentrations.

Nowadays most atomic spectroscopists follow either of two approaches for the determination of detection limit (4, 5): the SNR approach, which uses the signal-to-noise ratio (SNR); or the SBR-RSDB approach, which uses the signal-to-background ratio (SBR) and the relative standard deviation of the background (RSDB). Each approach is correct, and each yields the same detection limit. However, practical applications of the SBR-RSDB approach to assess conventional ICP systems using photomultiplier tubes (PMTs) as detectors (6-9) and advanced ICP systems based on array detectors (10-12) suggest that this approach is finding wider acceptance. The mathematical expression of this approach is as follows:

$$C_L = k \times 0.01 \times \text{RSDB} \times \frac{C_0}{\text{SBR}}$$

where:

RSDB is the relative standard deviation of the background (expressed as a percentage); SBR is the signal-to-background ratio; C_0 is the concentration which is yielding a net analyte signal; k is a constant of 2 or 3.

Toxic Heavy Metal Analysis of Cotton Gin Waste

Apparatus

Inductively coupled plasma atomic emission spectrometry

A Perkin-Elmer ICP/5500 (Perkin-Elmer Analytical Instrument, Norwalk, CT) with a Perkin-Elmer Model-10 data station and a Pr-100 printer were used for analyzing samples. The instrumental operating parameters together with the analytical lines chosen are given in Table 4.

Microwave Oven

A commercial microwave oven from Kenmore Inc., Model 566, Chicago, IL, was used for the acid digestion of sample. The oven has a variable timing cycle from 1 second to 100 minutes and a variable heating cycle based on power setting from 10% through 100% full power (700 w).

TABLE 4. ICP OPERATING PARAMETERS AND SAMPLE INTRODUCTION CONDITIONS

ICP operating parameters	
ICP-AES	Perkin-Elmer ICP/5500
RF power	1.25 Kw
Plasma gas	13 L/min.
Nebulizer gas	0.5 L/min.
Auxiliary gas	0.8 L/min.
Nebulizer pressure	12 psi
Viewing height	15 mm above load coil
Integration time	3 sec.
Sample introduction conditions	
Torch type	quartz torch demountable (Fassel type)
Nebulizer type	cross flow type
Spray chamber	Scott double pass type
Peristatic pump rate	0.5 mL/min.

Analytical lines

Element	Wavelength nm	Background correction
As	193.76	-0.07+0.06
Cr	205.55	-0.08+0.05
Pb	220.35	-0.05+0.05

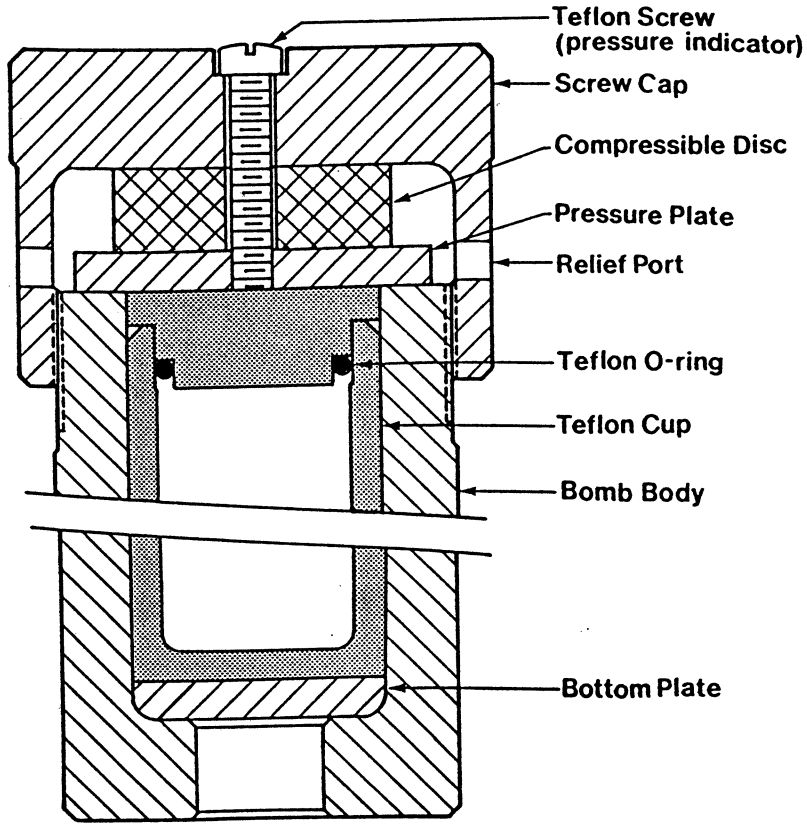
Parr Bombs

Parr teflon acid digestion bombs were obtained from Parr Instrument Co., Molin, IL. The bomb is made of a microwave-transparent polymer. The bomb includes several parts and is shown in Figure 5. A compressible relief disc built into the closure is designed to release excessive pressure before it reaches a level which might destroy the bomb and the oven. At approximately 1,500 psi the relief disc will be compressed to a point where support for the O-ring will be lost and the ring will blow out. In most cases all parts of the bomb except the O-ring will be reusable.

Reagents

All nitric acids used in this work were analytical reagent grade chemicals from Fisher Scientific Chemical Co., Houston, Texas. The water used to prepare all the solutions and analyze the sample was deionized water obtained by purifying the house deionized water with a Milliq system from Millipore Corp. This system produced a water of 18 MΩ/cm specific resistivity. An ICP-AES analysis of all of the water and acid used showed the presence of none of the three elements of interest in this study. Thus, if any

FIGURE 5. PARR TEFLON ACID DIGESTION BOMB



elements were present in it, they would be in sub-ppb amounts. The standard stock solution (1000 μg/ml) of As was purchased from Alfa Aesar Chemical Co., Wardhill, MA, and each stock solution (100 μg/ml) of Cr and Pb was prepared by dissolving a weighed portion of the high purity metal or salt (CrO_3 and $Pb(NO_3)_2$) in a dilute acid.

Sample Digestion Procedure

About 100 mg of sample were placed in a polyteflon (PTFE) container and treated with 2.5 ml of nitric acid. After the bottle was tightly capped, the bottle rack was placed in the microwave oven. The system was operated at full power for 30 seconds. After heating, the rack was removed from the oven and cooled by cold water for 30 minutes. When cool, the PTFE container was uncapped and 10 ml of deionized water was quickly added. The container was recapped and stood for 1 minute at room temperature.

At this stage, a very small amount of undigested residues still remained. The sample solution with the residue was filtered and washed with deionized water, and the filtrate was diluted to 50 ml in a volumetric flask.

Analytical Procedure

The analyzing instrument was calibrated using a calibration blank solution containing the same amount of

nitric acid, and single element standard solutions containing 10 ppm each of As, Cr, and Pb. Before beginning the sample run, the highest mixed calibration standard was reanalyzed as if it were a sample. The concentration values obtained did not deviate from the actual values by more than 5%. The analyzing system was flushed with the calibration blank solution for at least 1 minute before the analysis of each sample. After each 10 samples, the instrument check standard and the calibration blank were analyzed for a verifying purpose. Finally, the toxic heavy metal concentrations of the sample solutions were obtained.

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CHAPTER III

RESULTS, DISCUSSION AND CONCLUSION

Results and Discussion

The results of three heavy metals analyses are shown in Tables 6 through 9. A total of 28 samples were analyzed and the units of all data obtained were reduced from μg/ml to μg/g for our practical convenience. Table 5 shows the detection limits of the three elements for the ICP-AES used. Based on the detection limits, all of the metal concentrations were finally determined.

According to the ICP analysis, Arsenic (As), Chromium (Cr), and Lead (Pb) contents in all of the four constituent parts were significantly higher than expected. They range from 29.5 PPM to 785.9 PPM (μg/g). Noticeably, their distribution is completely random, presumably because of the heterogeneous nature of the all of the waste samples. Therefore, it is not assured that the three most toxic heavy metals will not cause any emission problem and/or will not exceed any EPA emission standards in the combustion of cotton gin waste as a fuel.

TABLE 5. DETECTION LIMITS* OF THREE ELEMENTS FOR ICP-AES

Element	Detection limit (μg/ml)
As	0.082
Cr	0.023
Pb	0.25

*: An integer of 2 was used for *k* constant, and 10 replicates of the background noise of each element were measured for the detection limit.

TABLE 6. HEAVY METAL CONCENTRATION OF BURS**

Sample No.	As	Cr	Pb
S1	360.0	530.0	360.0
S2	88.4	29.5	nd*
S3	314.6	190.7	390.9
S4	333.7	186.5	nd
S5	193.8	135.7	nd
S6	326.6	144.1	nd
S7	625.6	nd	nd
Average	320.4	173.8	-

*: nd - not detected.

**: Average of three replicates;
units - μg/g (PPM).

TABLE 7 HEAVY METAL CONCENTRATION OF STICKS**

Sample No.	As	Cr	Pb
S1	190.0	450.0	nd*
S2	137.1	127.3	283.9
S3	nd	30.0	nd
S4	413.8	443.4	nd
S5	nd	174.2	nd
S6	283.0	nd	nd
S7	609.9	nd	nd
Average	233.4	175.0	-

*: nd - not detected.

**: Average of three replicates;

units - μg/g (PPM).

TABLE 8. HEAVY METAL CONCENTRATION OF LINT**

Sample No.	As	Cr	Pb
S1	nd*	nd	nd
S2	125.2	134.8	317.8
S3	179.9	129.9	379.8
S4	280.1	130.7	nd
S5	206.6	281.7	nd
S6	131.7	nd	nd
S7	785.9	39.3	nd
Average	244.2	102.3	-

*: nd - not detected.

**: Average of three replicates;

units - μg/g (PPM).

TABLE 9. HEAVY METAL CONCENTRATION OF FINE DUST**

Sample No.	As	Cr	Pb
S1	nd*	nd	nd
S2	139.8	79.9	nd
S3	216.1	nd	648.3
S4	nd	287.7	257.9
S5	158.4	267.3	nd
2S6	612.8	136.2	nd
S7	757.4	nd	nd
Average	269.2	110.2	-

*: nd - not detected.

**: Average of three replicates;

units - µg/g (PPM).

During the separation procedure of cotton gin waste, maximum allowable particle size of fine dust was to be less than 4 mm. The percentage of burs, sticks, lint, and fine dust in each of 11 samples was determined and included in Table 10. This table shows that almost half of cotton gin waste consists of fine dust. This value is higher than the previously reported value of 33 % (1). It means that roughly a maximum of 0.6 million tons of the waste, excepting the fine dust portion, can be utilized as a fuel in Texas if the percentage data from only one gin is valid over all gins in Texas. Particularly, the weight percentage of the lint portion, which is of our interest is an average of 21.1 %. It reflects that Texas will produce about 0.21 million tons of lint and/or nearly 3.6×10^9 million Btus from lint waste each year (2).

Tables 11 through 15 show the moisture content of four constituent parts and of the raw material of cotton gin waste. All averaged values of moisture content have considerably good standard deviation. Finally, they were satisfied at 95 % confidence level. Among them, the lint portion has the lowest moisture content of averaged 7.26 % and the stick portion has the highest moisture content of 10.42 %. Therefore, lint was identified as within the Pellet Fuels Institute (PFI) standards of 8 % moisture

content requirement for a premium grade fuel. Average moisture content of the original raw cotton gin waste was calculated and examined, and they were found to show good agreement within an allowable error.

TABLE 10. WEIGHT RATIO OF FOUR CONSTITUENT PARTS OF CGW

Constituent / Sample part No.	Burs (wt. %)	Sticks (wt. %)	Lint (wt. %)	Fine dust (wt. %)	Total weight (g)
1*	25.9	8.6	16.7	48.9	49.0
2	26.5	2.5	15.7	55.4	12.4
3	20.0	1.7	9.2	69.2	12.4
4	17.8	6.2	24.8	51.2	13.1
5	24.1	8.1	23.0	44.8	8.8
6	34.5	2.6	21.6	41.4	11.8
7	24.0	6.6	9.6	59.9	16.8
8	29.4	9.4	29.4	31.8	8.6
9	29.3	10.2	17.4	42.9	16.8
10	26.8	8.9	28.6	35.7	11.2
11	17.1	8.9	35.8	38.2	12.4
Average	25.0	6.7	21.1	47.2	12.43
S.D. (σ)	5.2	3.0	8.2	11.1	-

*: Data of sample No.1 were excluded from the calculations of average and standard deviation because of an exceptionally large amount when compared to the others.

TABLE 11. MOISTURE CONTENT OF BURS

Sample No.	Initial mass, M (g)	Oven-dry mass, D (g)	Moisture content (%)
1	0.903	0.814	9.86
2	1.232	1.106	10.23
3	0.912	0.817	10.42
4	0.875	0.791	9.60
5	1.204	1.076	10.63
6	0.901	0.812	9.88
7	0.687	0.624	9.17
8	0.893	0.801	10.30
9	0.881	0.789	10.44
10	0.671	0.602	10.28
Average	0.916	0.823	10.08
S.D. (σ)	-	-	0.42

95 % CL = $\underline{10.08 \pm 0.26}$ % burs

TABLE 12. MOISTURE CONTENT OF STICKS

Sample No.	Initial mass, M (g)	Oven-dry mass, D (g)	Moisture content (%)
1	0.090	0.080	11.11
2	0.098	0.088	10.20
3	0.346	0.311	10.12
4	0.207	0.186	10.15
5	0.105	0.093	11.43
6	0.283	0.253	10.60
7	0.208	0.189	9.14
8	0.284	0.254	10.56
9	0.218	0.197	9.63
10	0.223	0.198	11.21
Average	0.206	0.185	10.42
S.D. (σ)	-	-	0.68

95 % CL = $\underline{10.42 \pm 0.42}$ % sticks

TABLE 13. MOISTURE CONTENT OF LINT

Sample No.	Initial mass, M (g)	Oven-dry mass, D (g)	Moisture content (%)
1	0.659	0.612	7.13
2	0.587	0.543	7.50
3	0.657	0.607	7.61
4	0.453	0.417	7.95
5	0.524	0.488	6.87
6	0.520	0.483	7.12
7	0.390	0.365	6.41
8	0.529	0.493	6.81
9	0.587	0.540	8.01
10	0.678	0.629	7.23
Average	0.558	0.518	7.26
S.D. (σ)	-	-	0.48

95 % CL = $\underline{7.26 \pm 0.30 \text{ \% lint}}$

TABLE 14. MOISTURE CONTENT OF FINE DUST

Sample No.	Initial mass, M (g)	Oven-dry mass, D (g)	Moisture content (%)
1	0.816	0.744	8.82
2	0.531	0.493	7.16
3	0.835	0.757	9.34
4	0.660	0.601	8.94
5	0.565	0.516	8.67
6	0.877	0.807	7.98
7	0.525	0.483	8.00
8	0.773	0.705	8.80
9	0.671	0.609	9.24
10	0.716	0.650	9.22
Average	0.697	0.637	8.62
S.D. (σ)	-	-	0.66

95 % CL = 8.62 ± 0.41 % fine dust

TABLE 15. MOISTURE CONTENT OF ORIGINAL CGW

Sample No.	Initial mass, M (g)	Oven-dry mass, D (g)	Moisture content (%)
1	0.894	0.826	7.64
2	1.199	1.100	8.22
3	1.025	0.945	7.74
4	0.962	0.887	7.79
5	1.547	1.403	9.30
6	1.469	1.338	8.90
7	1.293	1.183	8.47
8	1.186	1.093	7.78
9	1.126	1.033	8.30
10	1.570	1.439	8.38
Average	1.227	1.125	8.25
S.D. (σ)	-	-	0.52

95 % CL = 8.25 ± 0.32 % original CGW

Average moisture content of original CGW calculated = 8.82 % (no confidence limit allowable)

Ash content of each of three parts of cotton gin waste is listed in Tables 16 through 18. All of the APAR data in those tables were determined on an as received basis. On the other hand, every APOD was calculated from the APAR data and the moisture content of each corresponding sample. Lint portion showed a good standard deviation and the lowest ash content either in as received or oven-dry basis, mainly due to both good homogeneity and pure cellulosic composition of lint. Therefore, both APAR and APOD of lint were satisfied at 95 % confidence level and were very close to the PFI standards of 3 % ash content required for standard grade fuel.

TABLE 16. Ash content of burs

Sample No.	Initial mass, W_2 (mg)	Mass of ash, W_1 (mg)	APAR (%)	APOD (%)
1	258.9	14.6	5.64	5.09
2	268.9	26.6	9.89	8.88
3	257.9	16.7	6.48	5.84
4	237.5	17.5	7.37	6.60
5	269.2	16.9	6.28	5.68
6	248.6	26.7	10.74	9.60
7	241.2	19.0	7.88	7.10
8	251.1	23.5	9.36	8.50
9	252.2	17.5	6.94	6.22
10	244.1	11.1	4.54	4.07
Average	253.0	19.0	7.51	6.76
S.D. (σ)	-	-	1.87	1.68

TABLE 17. ASH CONTENT OF STICKS

Sample No.	Initial mass, W_2 (mg)	Mass of ash, W_1 (mg)	APAR (%)	APOD (%)
1	203.0	10.7	5.27	4.75
2	200.8	17.2	8.57	7.69
3	200.7	14.2	7.08	6.29
4	201.7	20.8	10.31	9.27
5	202.6	17.8	8.79	7.90
6	205.4	10.5	5.11	4.53
7	208.8	22.9	10.97	9.81
8	201.4	22.1	10.97	9.97
9	216.2	19.0	8.79	7.86
10	207.6	20.0	9.63	8.71
Average	204.8	17.5	8.55	7.68
S.D. (σ)	-	-	2.02	1.84

TABLE 18. ASH CONTENT OF LINT

Sample No.	Initial mass, W_2 (mg)	Mass of ash, W_1 (mg)	APAR (%)	APOD (%)
1	200.6	6.4	3.19	2.96
2	224.8	7.1	3.16	2.92
3	229.7	7.5	3.27	3.04
4	227.4	6.5	2.86	2.64
5	208.7	7.2	3.45	3.18
6	222.9	12.4	5.56	5.18
7	210.1	8.2	3.90	3.63
8	207.1	7.5	3.62	3.39
9	213.0	7.1	3.33	3.11
10	214.6	7.1	3.31	3.04
Average	215.9	7.7	3.57	3.31
S.D. (σ)	-	-	0.72	0.67

95 % CL = 3.57 ± 0.45 % APAR of lint.

95 % CL = 3.31 ± 0.42 % APOD of lint.

Conclusion

Consequently, lint, out of the three components of cotton gin waste, i.e., burs, sticks, and lint, will be the most prospective potential alternative fuel which most closely meets the most industrial fuel standards from Pellet Fuels Institute (3). On the other hand, heavy metal concentrations of lint as well as burs and sticks do not show a clear evidence of fulfilling the environmental emission standards of three of the most toxic heavy metals, e.g., Arsenic (As), Chromium (Cr), and Lead (Pb).

Therefore, it is required to investigate the heavy metal concentrations in ash left from the combustion of pelletized cotton gin waste fuel. Hence, the fate of heavy metals during the combustion will be very essential for determining whether or not cotton gin waste will cause some environmental emission problem. In addition, it also needs to be further investigated and/or developed in several aspects, e.g., an exact heat value of lint, analysis of organic emissions, pelletization-study of lint, more effective fine dust removal method, etc. After all, the results from the tasks suggested above will deeply influence the future of cotton gin waste as pellet fuel.

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3. Refer to Table 19 in Appendix

APPENDIX

SUMMARY OF PELLET FUEL STANDARDS FROM PELLET FUELS INSTITUTE

TABLE 19. SUMMARY OF PELLET FUEL STANDARDS FROM PFI

Fuel characteristics	Premium grade	Standard grade
Bulk density	≥ 40 lb/ft^3	≥ 40 lb/ft^3
Dimensions	Diameter 1/4 inch to 5/16 inch	Diameter 1/4 inch to 5/16 inch
British Thermal Units (BTU's)	≥ 8,200 BTU/lb	≥ 7,700 BTU/lb
Moisture content	8 %	8 %
Ash content	< 1 %	< 3 %
Fines	≤ 0.5 wt. %	≤ 0.5 wt. %

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