ELECTRON BEAM TECHNOLOGY FOR THE CONSERVATION OF CULTURAL HERITAGE MATERIAL

An Undergraduate Research Scholars Thesis

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ABSTRACT

Electron Beam Technology for the Conservation of Cultural Heritage Materials

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The development of non-invasive preservation techniques is an increasingly integral component in the field of cultural heritage conservation. Without proper storage and handling, the integrity of cultural artifacts materials can be lost. Electron beam technology has the potential to provide a novel approach to reduce the effects of mold degradation of valuable artifacts by providing a non-invasive preservation technique.

This study evaluated the capability of reducing microbial species with electron beam technology, without significantly altering the mechanical and physical properties of the material. Four varieties of paper were treated at target dose points of 0, 5 and 15kGy and subsequently observed for physical and mechanical changes with respect to each dose point. The aim of this study was to determine the appropriateness of electron bean decontamination of cultural materials as a preventative measure against losing these valuable artifacts.

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NOMENCLATURE

A. brasiliensis	Aspergillus brasiliensis
eBeam	Electron Beam
GC	Gas Chromatography
MeV	Million Electron Volts
DUR	Distribution Uniformity Ratio
kN	KiloNewton
kGy	KiloGray
SPME	Solid-Phase Micro-Extraction
CI	Confidence Interval

SECTION I

INTRODUCTION

The development of non-invasive preservation techniques is an increasingly integral component in the field of cultural heritage conservation. Both historical and cultural documents are composed of natural polymers susceptible to contamination and deterioration. Many species of fungi thrive and release airborne spores in warm temperatures with a relative humidity between 12 and 42% [1]. Not only can fungi found in libraries damage historical material, some molds can cause serious health issues due to their production of mycotoxins in addition to their ability to produce systemic infections [2]. While modest research has been completed with other preservation methods, such as gamma radiation sterilization, electrical beam processing is still a new process that needs to be explored [3].

Overall Objectives

Objective 1 of this experiment was to examine the effect of eBeam doses on color, olfactory and tensile properties. Objective 2 was to determine the presence/absence of A. brasiliensis on eBeam doses, but due to the unforeseen circumstances of COVID-19, that portion could not be completed. I hypothesized that high energy electron beam irradiation will not significantly alter paper materials, contributing to the preservation of cultural artifacts.

Rationale for Choice of Experimental Materials

Paper is a multi-component material, composed of wood-derived fibers such as cellulose, hemicelluloses and lignin [4]. Due to its complex composition, the susceptibility of paper to fungal deterioration can vary between materials. The difference in additives between papers can strongly influence the degradation process [5]. Therefore, the investigation of the degradation of various paper samples, such as ARCHES cotton, hemp drawing, HP photoprint and newsprint paper was established to determine the specific effects eBeam doses have on fungal contamination.

Background Literature

Ionizing radiation

With a wide selection of curative and preventative measures, such as fumigation and proper storage and handing, cultural heritage materials can still succumb to mold species, because of their ability to become active in the right storage conditions [6]. Classical methods of sterilization that have been borrowed from medicine and agriculture, like ethylene oxide (ETO) and methyl bromide (CH_3Br), are highly unpredictable and cannot be accurately controlled with cultural artifacts [7]. However, different ionizing radiation sterilization techniques such as gamma radiation and eBeam processing can be used for the sterilization of a variety of materials.

Gamma radiation is widely used in food, medical devices and pharmaceutical sterilization, and is characterized by the generation of photons from radioactive isotopes such as cobalt-60 and cesium-137. Gamma radiation has the ability to penetrate thicker and denser products at a greater capacity, whereas eBeam is best when a dose is delivered to thinner materials [6]. Previous research on paper disinfection has proven gamma radiation to maintain the physical properties of paper, but as an advanced technological method, this process has presented numerous disadvantages as alternative sterilization methods become available [8].

The electron beam sterilization method has quickly become a standardized practice for numerous medical devices and pharmaceuticals due to its effectiveness and harmlessness [6]. This continuous process is characterized by concentrated high speed electrons, generated by a linear accelerator, that cause reactions with molecules and microorganisms to render materials sterile. As a consequence of the scattering of electrons, the absorbed dose is delivered to the products and can be measured (Gy = 1 J/kg), with the minimum and maximum dose depending on the product itself. While the amount of dose the product receives can be controlled, certain physical characteristics such as color may be altered at higher doses. Ultimately, the choice of sterilization will depend on a variety of things, including the product specification, desired dose and the appropriateness of the process, and should only be selected based on the chemical and physical properties on the material. The eBeam process has the ability to reduce the impending degradation to polymers and materials due to the use of higher dose rates, which in turn will reduce the exposure time, and produce sterile products [3].

Biodegradation is responsible for the deprivation of multiple library archives, but can only be effectively reduced by exposing the material to liquid biocides and fumigation processes, which can be high toxic methods [2]. Most of the mold families responsible for deteriorating plant and animal substrates are saprophytes, and are capable of growing on most organic materials, especially cellulose [6]. The growth of these microorganisms depends on a variety of attributes, but with the appropriate amount of moisture in the environment, mold species can deteriorate important archival materials. Degradation of organic material, especially cellulose, is caused either by endogenous or exogenous factors [6]. Exogenous factors include altering the environment of the material, like the temperature and moisture, and can create a sustainable environment for fungal species to thrive. Electrons have the ability to damage DNA strands by producing hydroxyl radicals that join with O_2 and produce high levels of hydrogen peroxide within cells [3]. This method of sterilization can effectively halt the spread of fungal contamination at appropriate levels of absorbed dose, that does not severely deteriorate the material.

Implications of the Study

There are numerous advantages to eBeam technology compared to other sterilization methods. Not only does eBeam provide a well-controlled dose range to be achieved with directed temperature, but it also has ability to quickly apply a dose and protect the materials properties. It is also important to understand that current commercial products, such as food and medical devices, using this process have short lifetimes, while the preservation of cultural materials has to be highly sensitive in order to preserve it for many centuries. This study aims to quantify the mechanical properties of paper to determine if the quality of the sample is maintained.

SECTION II

MATERIALS & METHODS

Experimental Materials

Four different varieties of paper were used to examine the effects of eBeam on color, odor and tensile strength. These four varieties include; ARCHES cotton paper, newsprint paper, hemp drawing paper and HP premium plus photopaper. For the purpose of this experiment, these mediums were purchased from Amazon.com, Inc. (Seattle, WA).

ARCHES Aquarelle/ARCHES Watercolor paper, is prepared on a cylinder mold with only 100% cotton fibers. Commonly used by artists and in the creative arts industry, this paper is conserved with alkaline pH stabilizers, is acid free and contains no optical brightening agents [9]. Most importantly, this type of paper is treated with a fungicidal treatment to prevent the appearances of mold species, a common issue with organic materials.

Newsprint paper is still widely used around the world today, and while most still enjoy archiving articles, the need to conserve the papers' integrity becomes obvious. In this experiment, ULINE newspaper was utilized. Newspaper is not only extremely thin, but it is composed of deinked wood-pulp fibers. This allows the paper to have improved opaqueness and printing quality. As a popular paper choice for newspapers, this variety of paper is relatively inexpensive and suitable for printing conditions in newspaper manufacturing, but can also provide the appropriate medium for a fungal contamination.

Industrial hemp (*Cannabis sativa* L.) is used in a wide variety of products that range from paper to construction materials. Due to its fast-growing nature, hemp has numerous advantages over cotton, such as strength and production per acre. Hemp is a specific strain of plant in the family *Cannabis* that is characterized by the amount of Tetrahydrocannabinol (THC) that is secreted. Typical hemp plants are secreting less than 0.30% THC, where recreational marijuana can be anywhere from 6 to 40%. For the purpose of this experiment, Hemp drawing paper, manufactured by the Green Field Paper Company, composed of 25% hemp and 75% fiber was examined.

Lastly, HP Premium Plus Photo Paper was used to investigate effects of eBeam doses on its physical properties. Photographic paper is an acid and lignin-free glossy material composed of numerous fibers. Uniquely, this paper type is covered with a light-sensitive emulsion that creates a coated layer over the surface. This layer allows ink to penetrate into the paper and preserve images for longer periods of time.

Each variety of paper holds unique qualities that all contribute to faster manufacturing, improving preservation and enhancing production. Upon eBeam processing, three physical examinations were performed on each variety to determine the effect of eBeam processing on paper degradation.

Preparation of Materials for Experimental Studies

GC-Olfactory

A Gas Chromatography-Mass spectrometry instrument was utilized (Agilent model 6920, Santa Clara, CA) to determine the volatile odors associated with paper and eBeam doses. In order to increase the area in which odor can be detected, four 1-inch x 1-inch square swatches of each variety of paper were precisely cut using a paper cutter. The volume associated with the glass jars were capable of holding four swatches of paper, while having enough room for a solid-phase micro-extraction (SPME) portable field sampler to be inserted through the Teflon lid without touching the swatches. The SPME extraction method does not only provide better accuracy and decrease preparation time, but it also involves using a fused-silica coated fiber to effectively absorb and concentrate analytes on its surface [10]. After eBeam processing, the cluster of samples were immediately transferred into glass jars in order to preserve the volatiles associated with eBeam dose points.

Color

Color differences were detected by using a Konica Minolta Chroma Meter (CR-400, Ramsey, New Jersey), and involved the use of larger swatches due to the Chroma meter head width. Paper swatches were cut, using a paper cutter, in rectangles with the dimensions 3 x 2.5 inches. After processing, samples were measured by placing the chroma head directly on the swatch, for the L*, a* and b* values associated with each dose point.

Tensile strength

In order to prepare samples to be loaded into the Instron 1kN tensile test machine (Model 5943, Northwood, Massachusetts), paper swatches were cut shorter than the dimensions standardized by ASTM. This was ultimately due to the delicacy and efficiency of loading each sample into the tensile machine. Ten paper swatches, per variety of paper, were precisely cut into 1-inch x 7-inch rectangular shapes and arranged in stacks for processing. Upon measurement, samples were loaded into the tensile machine and measured for force of break in order to determine the tensile strength associated with each dose-point. Previous studies have shown a decrease in tensile strength when higher eBeam doses are applied to polymers [11].

Electron Beam Dose Delivery and Dosimetry

Electron beam processing was performed at the eBeam facility of the National Center for Electron Beam Research at Texas A&M University. A 10 MeV, 15kW linear accelerator delivered the eBeam doses. In order to confirm the delivered doses, alanine (L-α-alanine) pellet dosimeters were used. Paper swatches for the analytical examinations were placed on a cardboard processing tray in a flat position in order to ensure a dose uniformity ration (DUR) of one, and can be seen in Figure 1. Each sample swatch was sealed and processed at the appropriate speed to achieve target doses of 5 and 15kGy.

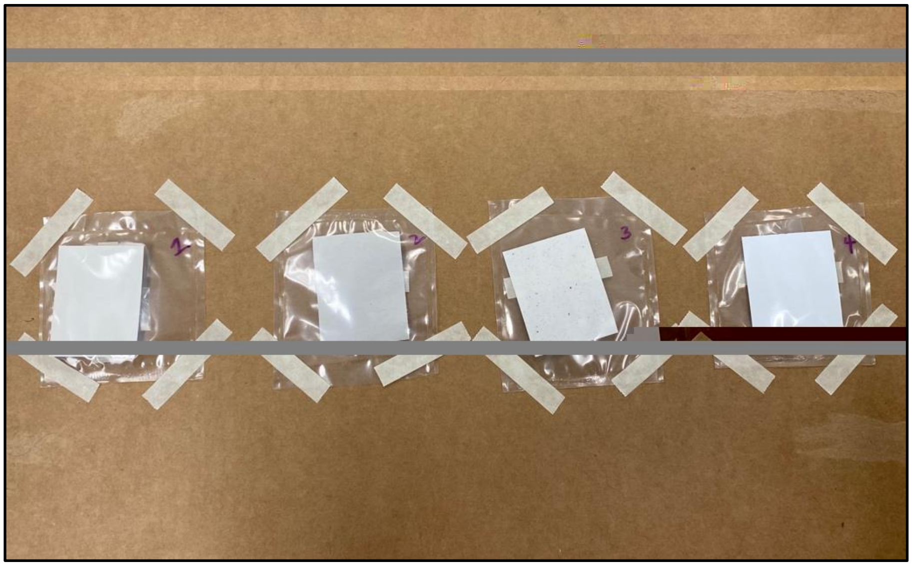

Figure 1. Example of paper swatches laid flat on cardboard processing tray

Analytical Methods

GC-Olfactory

In order to investigate the release of volatile odors due to eBeam processing, a Gas Chromatography-Mass spectrometry instrument (Agilent model 6920, Santa Clara, CA) was utilized. Post-processing, each paper sample was immediately inserted into glass jars. Each jar was heated to enhance the odors associated with each dose point, then the headspace was collected with a solid-phase micro-extraction portable field sampler. After the collection, the SPME was injected into an injection port where the sample was desorbed at 280°C. Samples were then loaded onto a multi-dimensional GC column, and then a second column. Next, the temperature was increased at a rate of 7°C/minute until reaching 260°C. The GC column then went to the mass spectrometer. This method provides a quantitative analysis of the chemicals that are released due to eBeam processing.

Color

To access any color dissimilarities, the CR-400 Chroma Meter (CR-400, Ramsey, New Jersey) was operated, by measuring the color differences using the parameters L*, a* and b*. L* refers to the amount of light and dark within an object, a* corresponds to red or green values and lastly, the b* values refer to how much blue and yellow pigments are in the material. The samples were measured for color differences in correspondence to the dose points 0, 5, and 15kGy, by placing the chroma head on the swatch. Values were given by the Chroma meter.

Tensile strength

Lastly, to identify the tensile strength of each paper sample post-irradiation, an Instron 1kN tensile test machine was used to provide a statistical measure of tensile changes between 0, 5, and 15kGy. In order to quantify (Model 5943, Northwood, Massachusetts) the average tensile strength, each swatch was loaded into the machine, in accordance with ASTM standards, placed in grips and elongated with the use of compressed air to determine the force at the break [12].

SECTION III

RESULTS & DISCUSSION

Color Changes Among Samples

Evaluating the differences in chromaticity coordinates L*, a* and b* gives an insight as to what color degradation is occurring due to eBeam doses. Due to the Prob>f, there is a statistical difference in L* mean values, in ARCHES cotton paper, across the treatment groups of 0, 5, and 15kGy at the 95% confidence interval (CI). There are also significant differences in L* mean values between 0 and 15kGy, where the lightness values have decreased at the higher dose. Little to no statistical differences were observed for the a* coordinate in cotton paper and are negligible. Significant changes in the b* coordinate can be seen in mean values between 0 and 15kGy, where the mean is increasing and directing towards more yellow values than blue.

L*, a* and b* color coordinates all differ in newsprint paper across the three dose points. Significant changes in the L* coordinate across treatment groups can be observed within means in the 95% CI. However, there is only a significant difference in 0kGy, when compared to the other dose point values. An increase in newspapers a* coordinate can also be detected, with major statistical differences between the three dose points.

Hemp drawing paper did not indicate any major statistically significant differences between 0, 5 and 15kGy. Although, there was a slight increase in the L* and a* coordinate, these changes are negligible and can be seen in Table 1.

However, HP Premium Plus Photopaper showed significant changes across all coordinates. The L* coordinate has minute differences, but show the mean values are different between each dose point, but do not increase nor decrease linearly. The a* coordinates display a significant increase in means at the 15kGy dose point. Lastly, the b* coordinates of photopaper indicate a difference in means between 5kGy and the other dose points, but no direct correlation is observed.

Table 1. Mean chromaticity coordinates among samples

Treatment:	**ARCHES Cotton Paper**		
	L*	**a***	**b***
0kGy	95.50^{ab}±0.056	-0.453±0.031	4.367^{ab}±0.091
4.93kGy	95.31^{a}±0.116	-0.460±0.017	4.523^{a}±0.067
14.69kGy	95.17^{ab}±0.056	-0.430±0.035	4.893^{ab}±0.137
Treatment:	**Newsprint Paper**		
	L*	**a***	**b***
0kGy	80.67^{ab}±0.095	1.107^{ab}±0.065	4.287^{ab}±0.168
4.93kGy	80.30^{a}±0.194	1.173^{a}±0.064	5.023^{a}±0.095
14.69kGy	80.08^{a}±0.026	1.257^{ab}±0.006	5.233^{a}±0.015
Treatment:	**Hemp Drawing Paper**		
	L*	**a***	**b***
0kGy	88.75±0.350	-0.560±0.036	3.733±0.080
4.93kGy	88.55±0.081	-0.563±0.051	3.743±0.397
14.69kGy	88.31±0.130	-0.603±0.021	3.917±0.107
Treatment:	**HP Premium Plus Photopaper**		
	L*	**a***	**b***
0kGy	94.39^{ab}±0.050	-0.537^{a}±0.006	-1.240^{a}±0.069
4.93kGy	93.65^{ab}±0.021	-0.527^{a}±0.006	-1.480^{ab}±0.061
14.69kGy	93.31^{ab}±0.050	-0.630^{ab}±0.020	-1.127^{a}±0.068

a. Signifies column mean values are significantly different ($P<0.05$).
b. Signifies mean values are significantly different between dose points.

Tensile Strength Changes Among Samples

Tensile strength differences for the different kinds of paper can be seen in Figure 2. and Table 2. Even with the average mean of ten samples, no direct correlation can be seen between dose points and tensile strength, other than in newspaper where there is an increase in strength at 15kGy. No changes were observed between 0 and 5kGy. These minute differences in tensile strength of paper are not significant and don’t change even at the highest dose point of 15kGy. These results are similar to observations made by other authors who also studied the effect of low absorbed dose values to tensile strength [13].

Table 2. Changes of tensile strength mean and standard deviation values

	Treatment:	0kGy	4.90kGy	15.39kGy
ARCHES Cotton Paper	Mean	6.417	5.606	6.102
	SD	0.028	0.055	0.031
Hemp Drawing Paper	Mean	4.764	5.079	4.921
	SD	0.030	0.006	0.010
Newsprint Paper	Mean	0.787	0.787	2.638
	SD	3.6571E-18	3.6571E-18	0.005
HP Premium Plus Photopaper	Mean	14.528	14.213	14.055
	SD	0.007	0.007	0.005

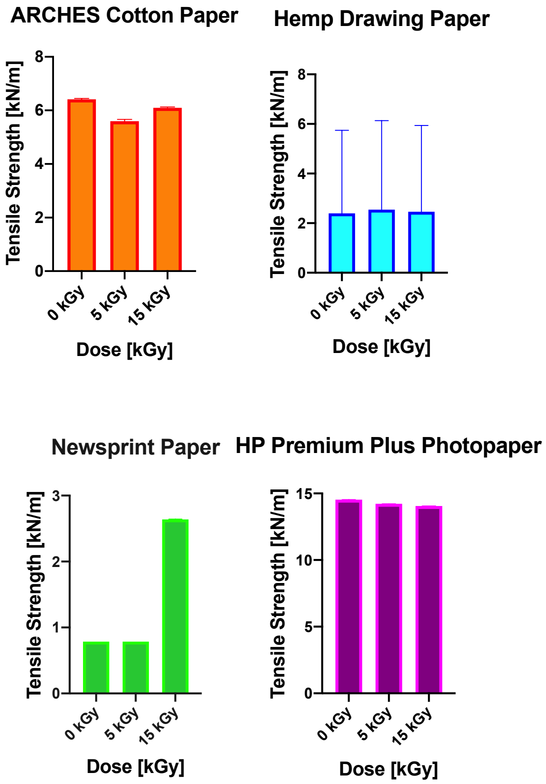

Figure 2. Changes of tensile strength among samples

Olfactory Changes Among Samples

Area values (ion counts) of relevant volatile odors, with an average area of more than 500,000, can be seen on Table 3. Every variety of paper contains unique manufacturing qualities that contribute to what chemical compounds are released as a result of eBeam doses.

ARCHES cotton paper displayed various volatiles, more than other types of paper, that are released as the dose increment increases. Benzene, (1,1-dimethylpropyl)-, benzene, 1,3-bis(1,1-dimethylethyl)- and nonadecane are all hydrocarbons that exclusively increased in area as the dose increment increased. Heneicosane demonstrated a rapid increase of area until it reached 15kGy and was no longer detected.

Hemp drawing paper displays a smaller quantity of volatile odors than that of cotton and photopaper. Similarly, the remaining papers also contain the hydrocarbons benzene, (1,1-

dimethylpropyl)- and benzene, 1,3-bis(1,1-dimethylethyl)- in large area percentages, which can be due to similar production methods of papers in general. Hexanal however, steadily increases as it the dose reaches 15kGy.

The volatile odors associated with newsprint paper also contained Benzene, (1,1-dimethylpropyl)- and benzene, 1,3-bis(1,1-dimethylethyl)-, but displays a large area of heneicosane at 15kGy. When compared to 0 and 5kGy, the area rapidly increases once a higher dose is applied.

Lastly, HP Premium Plus Photopaper, the most chemically altered type of paper contained large areas of the hydrocarbon benzene, (1,1-dimethylpropyl)- and benzene, 1,3-bis(1,1-dimethylethyl)-. Unlike newsprint paper however, eicosane rapidly decreased as the dose increased. As you can see in Table 3., eicosane was not detected in the higher dose. 2-propanone was also detected to have a higher area at 15kGy.

Table 3. Production of volatiles among samples

	ARCHES Cotton Paper			Hemp Drawing Paper		
Dose [kGy]	0 kGy	5 kGy	15 kGy	0 kGy	5 kGy	15 kGy
Area (ion counts)						
1-Acetoxy-2-propanol	120076	757325	385631	nd	nd	nd
1,2-Propanediol	340411	12037051	4296614	nd	nd	nd
2-Propanone	7529	1424075	4100038	nd	nd	15290
Acetone	nd	700548	nd	nd	nd	5857
Benzene, (1,1-dimethylpropyl)-	nd	359324	1040514	nd	38767	84234
Benzene, 1,3-bis(1,1-dimethylethyl)-	404652	550960	962980	575415	326972	1053294
Benzene, 1,4-dichloro-	774185	500593	1041507	538789	1723683	2211754
Dodecane	235506	78807	116776	244391	97891	346575
Eicosane	20657	7404	3427727	1968176	1928380	296240
Ethyl N,N-dimethylcarbamate	78323	1633425	508628	3885	nd	nd
Heneicosane	2109	16434	nd	2235	nd	nd
Hexanal	30634	nd	99169	913	7140	31362
Hexane	260529	81317	271073	292391	98321	463563
Nonadecane	29553	427156	1659538	15584	1715	12271
Octadecane	29548	8645	1348563	37363	7822	26156
Propylene Glycol	720344	2562572	1708282	nd	nd	nd

	Newsprint Paper			HP Premium Plus Photopaper		
Dose [kGy]	0 kGy	5 kGy	15 kGy	0 kGy	5 kGy	15 kGy
Area (ion counts)						
1-Acetoxy-2-propanol	nd	nd	nd	nd	nd	nd
1,2-Propanediol	nd	nd	nd	nd	nd	nd
2-Propanone	3137	nd	20590	2049	25238	102682
Acetone	nd	nd	nd	nd	nd	nd
Benzene, (1,1-dimethylpropyl)-	nd	nd	58927	114011	380681	1192366
Benzene, 1,3-bis(1,1-dimethylethyl)-	463563	538428	776889	85076	1127183	2975848
Benzene, 1,4-dichloro-	804583	1019265	931352	195333	545188	699016
Dodecane	451026	213340	144830	46846	34420	35027
Eicosane	13708	61625	812090	2728329	1779210	nd
Ethyl N,N-dimethylcarbamate	nd	nd	nd	nd	nd	nd
Heneicosane	13571	4045	1725794	nd	nd	2372
Hexanal	799078	454047	430340	17205	586	43444
Hexane	356763	301702	570778	475890	378774	411365
Nonadecane	833920	nd	4846	4917784	710831	395845
Octadecane	37402	29322	22773	2880592	20893	15560
Propylene Glycol	nd	nd	nd	nd	nd	nd

Elimination of Fungal Spores

Due to the unforeseen circumstances of the COVID-19 virus in spring 2020, complete data was unavailable at the time of publication for this URS thesis.

SECTION IV

CONCLUSION

Electron beam processing of cotton, hemp, newsprint, and photoprint paper show respective color changes between the chromaticity coordinates. While hemp drawing paper shows no major color changes, newsprint and cotton paper both display a minor decrease in lightness values compared to that of photopaper. These decreases in L* coordinates indicate the paper is becoming darker [5]. Most of the changes to color occurred within L* values, but differences in a* and b* coordinates are also observed. Especially in newsprint paper where the a* color coordinate indicates a more yellowish tone of color and b* coordinate denotes a more reddish tone is being produced. Color differences in HP photopaper are not as correlated, and provide no indication that eBeam doses are responsible for altering the chromaticity coordinates.

Evaluation of the mechanical properties of paper materials did not provide sufficient evidence to suggest a correlation between eBeam doses and tensile strength. The sharp increase of tensile strength in newspaper at 15kGy does not correlate to the other data associated with this variety of paper. Although the force of break varied between the ten samples measured per treatment group, the overall average means did not provide statistical evidence on the effect of eBeam doses on the mechanical properties of paper. Although studies have shown a correlation of eBeam doses with tensile strength and different materials, that association was not observed here [11].

Odor analysis specified volatile odors released when eBeam doses are applied. Although the manufacturing process of these papers may not be public, the majority of these chemicals associated with the paper samples come from plants and are known volatile odors [14-16]. The significance of this study was to provide the average areas of volatile odors associated with eBeam doses. In ARCHES cotton paper, benzene, (1,1-dimethylpropyl)-, benzene, 1,3-bis(1,1-dimethylethyl)- and nonadecane displayed an increase of area as the dose increment increased. Nonadecane specifically, is an alkane hydrocarbon composed of 19 carbon atoms, and widely used in essential oils that are isolated from the plant *Artemisia armeniaca.* The type of plant fibers used in the manufacturing of ARCHES cotton paper is unknown to most customers, but as a plant metabolite, nonadecane has been known to be a volatile odor component [14]. Heneicosane, another known volatile used to create essential oils, demonstrates a rapid increase of area until it reaches 15kGy, and is no longer detected. In hemp drawing paper, a smaller area of volatile odors was detected. Hexanal, a fruit flavored chemical commonly found in foods, displayed an increase in area as the dose increased. This chemical is widely used as a useful additive in the food science industry [15]. Heneicosane had a steady increase of area in newsprint paper, as the dose increased. Heneicosane once again is a volatile odor associated with the production of essential oils. Typically harvested from the plant *Carthamus tinctorius*, this compound can also have a variety of cosmetic applications [16]. Lastly, photopaper did not detect volatiles in larger areas different than those of the other varieties of paper, but did have a loss of eicosane as the dose increased. Further analysis on the composition of paper and the volatile odors associated with each variety can be further investigated, to determine if these volatile odors hinder the integrity of cultural heritage materials.

Furthermore, eBeam processed papers do not have a noticeable color change to the naked eye, but do show statistical differences among treatment groups. Mechanical testing does not provide statistical significance to determine if eBeam doses are associated with the decrease of tensile strength of paper. Further odor analysis can be completed to identity what odors are not naturally associated with archival materials, and if these volatile odors are responsible for degradation.

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