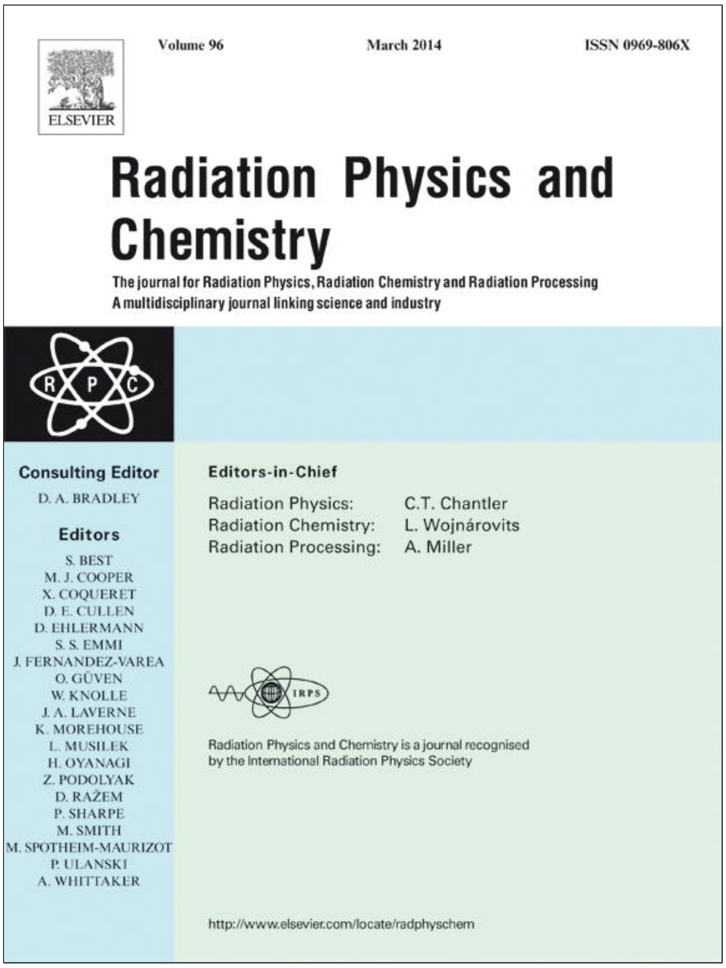

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Influence of dose and dose rate on the physical properties of commercial papers commonly used in libraries and archives

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HIGHLIGHTS

- Gamma irradiation is a valid option to remove mold from books and documents.
- We studied the effect of irradiation dose and dose rate on the physical properties of papers.
- We found an optimum combination of dose and dose rate.

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ABSTRACT

The aim of this study was to evaluate the effects of dose and dose rate of gamma irradiation on the physical properties of commercial papers commonly used in libraries and archives to optimize the irradiation conditions. Three different brands of paper of different fiber compositions were treated, using a 3^2 factorial design with four replicates of the center point, with doses ranging from 2 to 11 kGy and dose rates between 1 and 11 kGy/h. Chemical, mechanical and optical properties were determined on the samples. With some differences between the different kinds of papers, tensile strength, elongation, TEA, and air resistance were in general, unaffected by the treatment. The minimum loss of tear resistance and brightness were obtained with doses in the range 4–6 kGy at any dose rate for all three kinds of paper. These conditions are ideal to remove insects and sufficient to eliminate fungus.

1. Introduction

Biodeterioration is the most common source of degradation of files and books in tropical and subtropical countries. Their effect may vary depending on environmental conditions and substrate composition (Area and Cheradame, 2011). The microorganisms that feed on cellulose and other organic products are extremely dangerous for the material and must be separated, not only because it contaminates other books or documents, but also especially because it can cause severe health problems for restaurateurs, archivist or librarian. Early works about deterioration caused by microorganisms in books and scrolls and about fungi species date back the 19th century (Sterflinger and Pinzari, 2012). A review of the various biological agents that could cause deterioration of paper and possible intervention strategies can be found in the paper of Bankole (2010).

Ionizing irradiation has been used in the world to eradicate biodeterioration agents in libraries and archives after major floods. By means of this process, large quantities of library materials have been sporadically recovered (Magaudda and Adamo, 2010). Since gamma irradiation is highly penetrating, it allows the simultaneous treatment of large quantities of packaged books. If carried out using the correct dose, it does not harm the material. Its use is a valuable option compared to the chemicals used so far (fungicides and other chemicals), which have proven to leave residues which are toxic and harmful to the humans and environment (Magaudda, 2004). Additionally, as it leaves no residue, library materials can be used immediately after being treated. Several articles refer to the dose required to eliminate the causes of biodeterioration (Sinco, 2000; Adamo et al., 2001; Adamo et al., 2004; Adamo et al., 2007). Recent investigations have focused on the study of the effects of irradiation on the properties of paper, to find the doses at which paper properties are affected as little as possible (Gonzalez et al., 2002; Calvo, 2004; D'Almeida et al., 2009; Calvo et al., 2010).

Abbreviations: AR, Air resistance; B, ISO brightness; CV, Coefficient of variation; D, Density; E, Elongation; SD, Standard deviation; T, Tint; TEA I, Tensile energy absorption index; Tear I, Tear index; Th, Thickness; TI, Tensile index; V, Intrinsic viscosity; W, Whiteness

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The aim of this study was to evaluate the effects of dose and dose rate acting in combination, on the chemical, physical, mechanical and optical properties of commercial papers commonly used in archives and libraries, in order to optimize the irradiation conditions for each of the studied kinds of paper. The hypothesis is that a dose may produce a different level of deterioration in the paper according to the dose rate at which such dose was absorbed. For doing this, papers of three different compositions were irradiated, evaluating the variation of viscosity, tear index, tensile index, elongation, TEA, air resistance, ISO brightness, whiteness, tint, and CIELAB color space parameters (L^*, a^*, b^*), after the application of different combinations of dose and dose rate.

2. Materials and methods

2.1. Materials

The evaluation included the response to irradiation of three different brands of paper with different pulp composition: (A) soda-anthraquinone pulp from sugarcane bagasse (Autor), (B) bleached eucalyptus kraft pulp with an elemental chlorine free bleaching sequence (Boreal), and (C) a specialty paper with a 25% of cotton fiber in use for paper conservation (Capitol Bond). The samples of the first two kinds of papers were taken at different positions of commercial reams and the third, in the same way from sheets.

2.2. Methods

The samples were irradiated in the Semi-Industrial Irradiation Plant of the National Atomic Energy Commission (CNEA), located at the Ezeiza Atomic Center.

The experiences were organized according to a 3^2 factorial design with 4 replicates of the center point (Fig. 1), with doses ranging between 2 and 11 kGy and dose rates from 1 to 11 kGy/h.

The evaluation included: intrinsic viscosity (V), thickness (Th), basic weight, density (D) tear index (Tear I), tensile index (TI), elongation (E), tensile energy absorption Index (TEA I), air resistance (AR), ISO brightness (B), whiteness (W), tint (T) and color parameters in the CIELAB space (CIE 1976) L^*, a^*, b^*: The TAPPI standards were used in for the measurement de mechanical and physical properties and ISO standard for the optical properties.

For comparative purposes, the color-difference (ΔE^*) can be calculated from the parameters L^*, a^*, and b^*, using the Eq. (1) (CIE, 2001):

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (1)$$

In which all differences were calculated from the parameters of the color of the untreated paper.

The inherent variability of non-irradiated papers was assessed using the coefficient of variation (CV). The CV is a dimensionless measure, which is used to compare the dispersion or variability between samples or distributions. It is the ratio between the standard deviation (SD) and the mean [(SD/mean)*100 (%)], and represents the existing deviation from the mean of a set of observations.

The results of the application of the experimental design were analyzed using multifactorial analysis of variance (ANOVA), multiple range test, and multiple regression analysis using Statgraphics software at 0.05% significance.

3. Results and discussion

3.1. Physical properties of the original (untreated) papers

The conditioned basic weights and the intrinsic viscosity of the original (untreated) papers were 80 g/m² and 676 mL/g for paper A; 80 g/m² and 695 mL/g for paper B; and 89 g/m² and 794 mL/g for paper C, respectively.

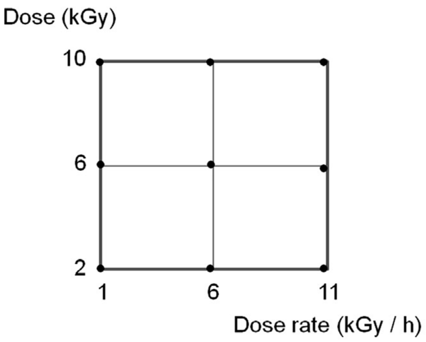
In order to verify the inherent variability of the papers under study, we first determined the properties of the three kinds of paper, extracted from different positions of reams (top, middle and bottom).

3.2. Properties of the irradiated papers

The properties of papers that showed significant variation with the different levels of the experimental design are shown in Table 1.

The inherent variability of the non-irradiated and irradiated papers was evaluated by the coefficient of variation (CV) of each property. Means and CV of all properties for non-irradiated and irradiated papers are shown in Table 2.

Comparing the variability of mechanical and optical properties of non-irradiated papers with the results obtained by applying the different irradiation conditions (Table 2), we conclude that neither the dose nor the dose rate have produced significant alteration of the mechanical properties of the three kinds of paper at the studied levels. For example, in the case of paper “Author”, "Autor" the inherent variability of tensile index (CV: 3.8%) was



Variables (real values)		Variables (coded values)	
Dose	Dose rate	Dose	Dose rate
2	1	-1	-1
10	1	1	-1
2	11	-1	1
10	11	1	1
2	6	-1	0
10	6	1	0
6	1	0	-1
6	11	0	1
6	6	0	0
6	6	0	0
6	6	0	0
6	6	0	0

Fig. 1. Experimental points of the 3^2 factorial design with four replicates of the center point.

Table 1
Viscosity, mechanical and optical properties of the irradiated papers A, B and C.

	Dose	(kGy)	2	10	2	10	2	10	6	6	6	6	6	6
	Dose rate	(kGy/h)	1	1	11	11	6	6	1	11	6	6	6	6
A	V	(mL/g)	478	292	549	348	473	290	406	359	370	378	370	369
	D	(g/cm³)	0.758	0.748	0.744	0.744	0.759	0.765	0.758	0.767	0.765	0.765	0.765	0.768
	Tear I	(mN m²/g)	4.94	4.53	4.92	4.57	5.03	4.68	4.85	4.75	4.74	4.7	4.68	4.74
	B	(%)	93.7	91.9	93.9	91.9	93.2	91	93	92.2	92.4	92.3	92.3	92.3
	b^*		−7.29	−6.00	−7.48	−6.15	−6.93	−5.4	−6.9	−6.23	−6.61	−6.26	−6.28	−6.28
	T		1.7	1.48	1.71	1.43	1.66	1.36	1.77	1.59	1.6	1.63	1.57	1.53
	ΔE^*		0.73	2.02	0.54	1.87	1.09	2.63	1.12	1.80	1.41	1.77	1.74	1.74
B	V	(mL/g)	524	311	542	349	535	298	409	393	359	359	370	393
	D	(g/cm³)	0.727	0.723	0.723	0.735	0.731	0.721	0.727	0.745	0.736	0.737	0.734	0.737
	Tear I	(mN m²/g)	7.57	7.26	7.66	6.95	7.73	7.16	6.9	7.44	7.25	7.25	7.27	7.22
	B	(%)	93.7	91.7	93.8	91.5	93.5	90.9	92.5	92.1	91.8	91.8	92	92.3
	b^*		−7.05	−5.83	−7.08	−5.59	−6.58	−5.19	−6.27	−5.95	−5.88	−5.88	−5.93	−6.06
	T		1.88	1.79	1.77	1.6	1.89	1.66	2.1	1.85	1.93	1.8	1.85	1.67
	ΔE^*		0.40	1.64	0.39	1.87	0.90	2.28	1.22	1.51	1.60	1.58	1.54	1.39
C	V	(mL/g)	525	314	541	348	537	299	446	386	427	444	405	379
	D	(g/cm³)	0.711	0.704	0.709	0.706	0.731	0.699	0.713	0.718	0.727	0.71	0.71	0.725
	Tear I	(mN m²/g)	9.67	8.34	8.91	8.89	9.44	8.51	8.64	8.71	8.85	8.97	9.11	8.51
	B	(%)	92.8	91.9	92.3	91.5	92	91.1	92	91.5	91.8	91.4	91.4	91.4
	b^*		0.51	0.88	0.62	1.07	0.96	1.40	1.05	1.16	0.93	1.23	1.25	1.28
	T		−0.3	−0.4	−0.3	−0.34	−0.33	−0.4	−0.28	−0.36	−0.33	−0.33	−0.31	−0.36
	ΔE^*		1.43	1.80	1.54	2.00	1.88	2.33	1.97	2.09	1.86	2.15	2.17	2.20

Table 2
Means and coefficients of variation of the mechanical and optical properties of the non-irradiated and irradiated papers.

		Th (mm)	D (g/cm³)	Tear I (mNm²/g)	TI (N m/g)	E (%)	TEA I (J/g)	AR (s)	B (%)	W (%)	L^*	a^*	b^*	T
A	Mean non-irradiated	0.106	0.754	5.14	57.9	1.43	0.54	51.1	94.6	119	92.8	0.71	−8.01	1.92
	CV % non-irradiated	0.9	1.3	3.1	3.8	9.3	13.2	11.0	0.5	0.59	0.2	9.9	1.7	7.3
	Mean irradiated	0.106	0.759	4.76	57.0	1.34	0.48	52.5	92.5	113	92.9	0.55	−6.48	1.59
	CV % irradiated	1.2	1.2	3.1	2.8	4.8	6.4	6.0	0.9	2.3	0.04	14.6	−9.0	7.6
B	Mean non-irradiated	0.108	0.740	7.50	76.2	2.18	1.12	12.5	94.3	117	93.0	0.46	−7.45	2.09
	CV % non-irradiated	0.9	0.2	5.9	4.6	6.3	6.3	10.1	0.2	0.30	0.1	7.25	1.1	1.7
	Mean irradiated	0.109	0.731	7.31	76.4	2.16	1.10	12.4	92.3	111	93.0	0.32	−6.11	1.82
	CV % irradiated	0.001	0.9	3.6	3.3	2.4	3.3	5.8	0.9	2.3	0.1	39.1	−8.8	7.4
C	Mean non-irradiated	0.124	0.717	9.19	53.8	2.49	0.92	17.9	92.2	89.0	97.2	0.04	0.92	−0.40
	CV % non-irradiated	1.1	0.4	3.0	2.6	4.4	6.4	6.1	0.1	0.19	0.04	58.3	4.3	6.7
	Mean irradiated	0.125	0.714	8.88	52.9	2.38	0.87	18.2	91.8	88.1	97.1	−0.03	1.03	−0.34
	CV % irradiated	1.2	1.4	4.3	2.3	2.5	3.8	7.1	0.5	1.6	0.07	−213	25.8	−11.3

higher than the CV value of the tensile index of the irradiated papers (CV: 2.9%), considering all points of the design. These results agree with those of other authors who studied the response of the tensile strength to low doses (Moise et al., 2012). By contrast, most of the optical properties of the irradiated papers showed greater variability due to different combinations of dose and dose rate.

3.3. Models of variation of the properties with respect to the variables studied

The equations corresponding to the variation of the properties of the three kinds of papers with respect to the variables studied (dose and dose rate, as transformed variables) are presented in Table 3 (the equations that were not significant are not shown).

Some authors claim that irradiation is restricted because it can produce cumulative depolymerization of cellulose and severe aging (Sterflinger and Pinzari, 2012; Ponta, 2008). The viscosity of the three kinds of paper decreases with the dose (Eqs. (2, 3 and 4)). In paper B, the variation with the dose has a slight quadratic effect, which means a steepest initial decrease. According to the findings of this work, the irradiation with 2 kGy–11 kGy/h produced the lowest viscosity reduction (19%, 22% and 32% for papers A, B and C respectively). Paper C, with cotton fibers, was the most susceptible to irradiation.

The tear index – critical in the case of documents and books – was the only mechanical property affected by the irradiation at the studied levels (from 2 to 10 kGy). The tear index of paper A (bagasse), decreased linearly with the dose and it was not affected by the dose rate (Eq. (5), model similar to that of viscosity), whereas paper B (eucalyptus) behaved in a similar way but with a steepest decrease (Eq. 6). The behavior of paper C was, in general, different from the other two, possibly due to the influence of cotton fibers (Eq. (7)). In the case of tear index, it is preferable to apply high dose rates when applying high doses. The decrease of tear strength was reported previously by other authors who worked with doses of 10 kGy and dose rates of 15.6 kGy/h (Butterfield, 1987).

Brightness quantifies the amount of blue light reflected by the surface of the paper at an effective wavelength in the blue region of the spectrum (457 nm). By definition, the CIE Whiteness of

Table 3
Regression equations for mechanical and optical properties as a function of dose and dose rate (in coded values of the variables).

Paper	Equation		Correlation coefficient
A	$V=375-95{*}\text{Dose}+28{*}\text{Dose rate}+45{*}\text{Dose rate}^2$	(2)	$R^2=99$
B	$V=380-107{*}\text{Dose}+46{*}\text{Dose}^2$	(3)	$R^2=95$
C	$V=428-107{*}\text{Dose}$	(4)	$R^2=96$
A	$\text{Tear I}=4.78-0.17{*}\text{Dose}$	(5)	$R^2=84$
B	$\text{Tear I}=7.33-0.26{*}\text{Dose}$	(6)	$R^2=77$
C	$\text{Tear I}=8.90-0.38{*}\text{Dose}+0.33{*}\text{Dose}{*}\text{Dose rate}$	(7)	$R^2=78$
A	$B=92.2-1.00{*}\text{Dose}+0.52{*}\text{Dose rate}^2$	(8)	$R^2=93$
B	$B=92.1-1.10{*}\text{Dose}+0.48{*}\text{Doserate}^2$	(9)	$R^2=94$
C	$B=91.5-0.43{*}\text{Dose}-0.23{*}\text{Dose rate}+0.48{*}\text{Dose rate}^2$	(10)	$R^2=87$
A	$W=112-3.17{*}\text{Dose}+1.73{*}\text{Dose rate}^2$	(11)	$R^2=90$
B	$W=110-3.27{*}\text{Dose}+1.61{*}\text{Dose rate}^2$	(12)	$R^2=94$
C	$W=87.4-1.10{*}\text{Dose}+1.38{*}\text{Dose rate}^2$	(13)	$R^2=71$
A	$T=1.59-0.13{*}\text{Dose}-0.09{*}\text{Dose}^2+0.084{*}\text{Dose rate}^2$	(14)	$R^2=85$
B	$T=1.91-0.08{*}\text{Dose}-0.09{*}\text{Dose rate}-0.14{*}\text{Dose}^2$	(15)	$R^2=81$
A	$\Delta E^{*}=1.79+0.69{*}\text{Dose}-0.45{*}\text{Dose rate}^2$	(16)	$R^2=91$
B	$\Delta E^{*}=1.58+0.69{*}\text{Dose}-0.41{*}\text{Dose rate}^2$	(17)	$R^2=95$
C	$\Delta E^{*}=2.23+0.21{*}\text{Dose}-0.21{*}\text{Dose}^2-0.28{*}\text{Dose rate}^2$	(18)	$R^2=90$

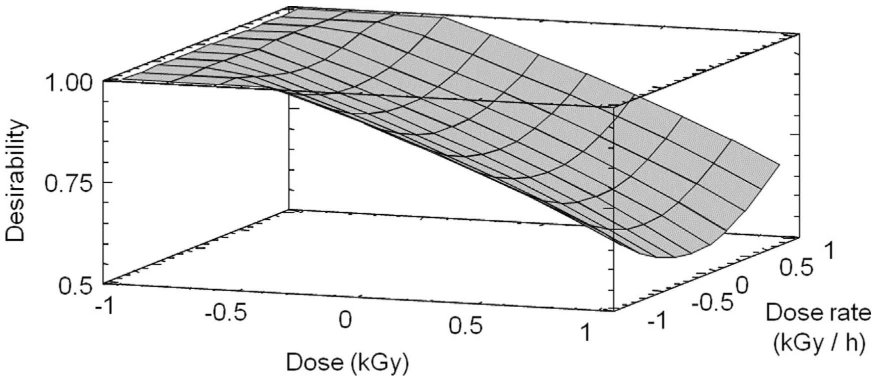


Fig. 2. Desirability function resulting from the equations of tear index, and brightness, for the optimization of dose and dose rate of paper C.

paper represents the visual sensation of white. It is the best predictor of the "white color" of a paper. Whiteness values above 100 generally indicate the presence of optical brighteners, as observed in the papers A and B (Table 1). The brightness decreased linearly with the dose showing a slightly quadratic behavior with dose rate in all cases (Eqs. (8, 9, and 10), model similar to that of viscosity), indicating that the greatest loss of this property occurs at the mean value (6 kGy/h). Again, paper C was the most affected by the dose rate. The whiteness showed a similar behavior (Eqs. (11, 12 and 13)); although in this case, the equations of variation in the three kinds of paper were quite similar. Tint indices are biased in the red–green dimension and describe the amount of greenish or reddish tint in products that are close to perfect white. Negative tint values indicate a reddish cast (slightly positive a*), while positive tint values indicate a greenish cast (slightly negative a*), (HunterLab, 2008). The tint decreased quadratically with the dose rate in papers A and B (Eqs. (14 and 15)) whereas these factors did not affect significantly the tint of paper C.

CIELab color space describes color components in relation to human vision. A paper "pure white" would have the parameters $L^{*}=100$, $a^{*}=0$ and $b^{*}=0$. The value of L^{*} represents the lightness (0 = black, 100 = white). The variation of L^{*} was minimal with the treatments, whereby equations were not significant for any paper. The color parameter a^{*} represents the change from green (−) to red (+). It decreased with the dose at any dose rate in papers A and B, whereas a^{*} values were close to zero (neutral) in paper C, in all points of the design. The parameter b^{*} denotes the change from yellow (+) to blue (−). In the case of papers A and B, its value was negative, i.e. bluish. The color of the paper C was initially neutral, and become increasingly yellow with the increase of the dose (positive b^{*}). The color-difference, increased linearly with the dose in the papers A and B (Eqs. (16, 17)), but in paper C, the dose produces an increase in the color difference only to a value of 8 kGy (Eq. (18)). In all cases, a dose rate of 6 kGy/h produced the maximum color-difference.

3.4. Optimization of the dose and dose rate

An optimization system of multiple equations (desirability function) was applied, combining the equations obtained for tear index and brightness. This approach to simultaneously optimize multiple equations, translates the functions to a common scale [0, 1], and combines them using the geometric mean and optimizing the overall metric. The minimum loss of tear resistance and brightness were obtained with doses in the range 4–6 kGy for all three kinds of paper at any rate. The representation of the desirability function for paper C is shown as example in Fig. 2.

The results of this work showed that the studied papers did not suffer appreciable alteration of air resistance, tensile strength, elongation and TEA, using doses between 2 and 10 kGy, and dose rates between 1 and 11 kGy/h. The variation of other properties when irradiating within these limits of dose and dose rate can be obtained from the regression equations. For example, if papers with historical value infected by microorganisms require a dose of 10 kGy, and considering a dose rate of 11 kGy/h, the decrease in tear index and brightness will be of 3.6% and 0.6% for paper A, 3.5% and 0.6% for paper B, and of 0.7% and 0% for paper C.

The strength of paper is determined by the intrinsic strength of fibers and by the strength of interfiber bonding. The property most influenced by the bonding is the tensile strength. Since the studied levels of irradiation did not change significantly this property in all papers, it can be inferred that the relative bonded area between fibers and the bond strength per unit of bonded area are not affected by irradiation. Beyond the possible weakening of interfiber bonds (since there is no evidence that), the strength of paper decreases because of the loss of intrinsic strength of fibers due to cellulose deterioration. Cellulose degradation is usually characterized by the decrease of its degree of polymerization and the molar mass, and both are related with the intrinsic viscosity (Area and Cheradame, 2011). The equations found in this work indicate that the variation can be different in different types of paper and it is clear that this effect is not directly proportional on the reduction of most mechanical properties, at least in the dose range studied in this work.

Studies about changes in the supramolecular structure of cellulose indicate that its degree of crystallinity slightly decreased using doses from 20 kGy to 120 kGy (Kasprzyk et al., 2004). Moreover, Kusama et al. (1976) found that the degree of polymerization of the fibers decreased with increasing dose, but their microcrystalline celluloses were only slightly degraded by irradiation, especially in microcrystalline cellulose from cellulose I. Consequently, the authors inferred that cellulose molecules in the amorphous regions are degraded more readily, and the well-aligned molecules in crystalline regions are not as easily degraded by irradiation. This could be the explanation for the lack of proportionality between viscosity loss and decreased tear resistance. This fact confirms that viscosimetry, although still widely used in cellulose analysis, should be replaced by size-exclusion chromatography, which allows the characterization of the molar mass distribution of the polymer, and provides information about the degraded fractions, thus leading to insights in the degradation mechanisms (Dupont and Mortha, 2004).

The preconception that irradiation depolymerizes and oxidizes the cellulose at levels as to generate a substantial loss of strength and optical properties generally comes from tests performed with extremely high doses, well above those used for removal of microorganisms. For example, Bouchard et al. (2006) found linear relationships between the number of chain scission in cellulose and the dose as well as between chain scissions and zero-span breaking length, but using 250 kGy to treat mail by the US Postal Service.

When there is an insect massive attack with no associated fungal development, the convenience of irradiation of the material is more evident. Required doses to remove insects are 0.5-1 kGy (Calvo, 2004), thus doses of 4-6 kGy (optimal range obtained in this work) are more than enough for this purpose. Moreover, the literature suggests that a dose of about 5 kGy is enough to inactivate fungi (Calvo, 2004; Dasilva et al., 2006). According to the found equations, the use of 5 kGy did not produce significant changes on the observed properties, and did not impair the normal use of documents and books treated with gamma irradiation. When the fungal attack is severe, the doses of usual application are 10 kGy. Differences between irradiating with 10 kGy and irradiating with the optimal doses and dose rates, implies a loss of tear strength of 10% approximately for all papers. However, it is noteworthy that the effects of irradiation on the properties of interest were negligible compared to the biodeterioration caused by the action of microorganisms on books and documents comprising the cultural heritage. This is confirmed by the experience of the library of the CNEA, in which documents declared as Memory of Humanity have undergone irradiation at a dose of 9 kGy and are in normal use.

4. Conclusions

This work shows that the use of experimental designs applied to conservation studies is a useful tool to identify the combination of variables that minimizes the deterioration caused by irradiation. The minimum loss of tear resistance and brightness were obtained with doses in the range 4–6 kGy at any dose rate for all three kinds of paper. Within these conditions, this treatment produced no critical changes in physical properties of the papers, so it can be said that regular use of documents and books would not be affected.

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