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Physicomechanical evaluaction of a Kraft pulp of "Pinus Pseudostrobus Lindl" After Bleaching With Peroxide Ultraviolet Light

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Abstract

In the present study, the factors and interactions affecting the degree of delignification, brightness level, and various physico-mechanical properties of Kraft pulp were analyzed by subjecting it to ultraviolet (UV) light irradiation as a bleaching method in the presence of hydrogen peroxide. A 2^3 factorial experimental design was carried out, where the factors were irradiation, peroxide concentration, and pH. The objective was to identify possible interactions among these factors and determine any adverse effects on pulp properties.

The analysis of variance (ANOVA) revealed that ultraviolet light has a significant effect on brightness and the kappa number, confirming findings previously reported by other authors. Furthermore, the statistical analysis showed that UV light does not significantly affect viscosity measurements or physico-mechanical properties of the pulp.

Introduction

High-purity cellulose has shown increasing demand not only in the paper industry but also in the food, cosmetic, and pharmaceutical sectors. This demand has driven the development of more efficient processes for its production and refinement. Although wood-based products remain the most viable source of cellulose fiber, their processing presents challenges, particularly in removing contaminants inherent to the lignocellulosic nature of the material (Ek et al. 2009).

One of the key characteristics of high-purity pulp is its brightness, as it indicates a significant removal of residual lignin after the separation of cellulose and lignin. However, brightness is not always a reliable indicator, since chromophoric compounds can remain in the cellulose and cause discoloration over time—a phenomenon known as color reversion (Andmdy et al. 1991). Therefore, it is necessary to complement brightness measurements with the kappa number, which provides an indirect assessment of the degree of pulp delignification (Cadena et al. 2010).

There are currently various methods for cellulose pulp delignification, notably the Elemental Chlorine Free (ECF) and the more desirable Totally Chlorine Free (TCF) processes, which offer reduced environmental impact (Ouchi et al.2003). These methods aim to achieve pulp quality comparable or superior to traditional technologies while minimizing ecological damage.

Tsuji et al. (2002) proposed an acid treatment followed by ultraviolet (UV) light exposure to remove hexauronic acid, which degrades into 2-furancarboxylic acid (FA) and 5-formyl-2-furancarboxylic acid—compounds responsible for color reversion. Their approach effectively reduced reversion by eliminating chromophoric groups, achieving results similar to elemental chlorine technologies, which did not present such reversion issues. These authors achieved brightness levels above 88\% without reversion, indicating very high cellulose purity with minimal use of harsh chemicals. This effect is attributed to the action of perhydroxyl and superoxide radicals, which can break lignin chains without significantxly damaging carbohydrate chains (An 1998, Gierer 1997, Liu 2009)

Literature reports that cellulose degrades five to six times more slowly than lignin when exposed to UV light in the presence of hydroxyl radicals (Ek et al. 2009). However, the interaction of variables such as pH, the concentration of radical-generating agents, and the wavelength of light complicates the analysis of these reactions and their combined effect on lignin and cellulose.

Ek et al. (2009) were among the first to propose that UV light could induce the generation of radicals to facilitate delignification and improve pulp brightness. Subsequently, Machado et al. (1996) reported the use of UV light in an oxidizing medium for bleaching purposes. Pérez et al. (1998) explored the use of titanium dioxide as a photocatalyst to generate hydroxyl radicals for treating unbleached pulp, although with limited efficiency.

Ouchi[2] et al. 2008) revisited this concept and obtained promising results using KrF laser light (wavelength of 248 nm) in a medium containing sodium borohydride. Later, his team experimented with various irradiation sources (KrF, XeCl, XeF excimer lasers, mercury lamps, and blue fluorescent lamps) in aqueous solutions of $Na_2CO_2 \cdot 1.5H_2O_2$ and H_2O_2 at different concentrations and combinations, designed to enhance photolysis without damaging the fibers. Their results showed that the photocatalytic process can achieve brightness levels comparable to traditional methods, with environmental advantages and potentially less fiber degradation. Through this research, they achieved brightness levels between 80 and 90 ISO (Ouchi[2] et al. 2008).

Additional tests were conducted using other light sources, such as blacklight lamps, in alkaline hydrogen peroxide solutions, also achieving brightness levels comparable to commercial pulps. Kurosu et al. (2009) used ozone as the oxidizing agent and achieved ISO brightness values ranging from 86% to 91%, surpassing conventional methods by 26 points.

In another line of research, Castellan et al. (1999) employed photosensitizers to enhance light absorption. The most effective compounds included methylene blue (MB), 3,4,9,10-perylenetetracarboxylic acid (PTCA), and iron (II) tetrasulfophthalocyanine (TSPC). These compounds, in combination with TiO_2 and UV or visible light, transferred energy to reactive oxygen species such as singlet oxygen or radicals that selectively attacked and degraded lignin. This approach increased bleaching efficiency, reduced the use of traditional chemicals and reaction time, and improved process selectivity, resulting in whiter and higher-quality pulp.

However, recent studies by Eren (2008) have shown that excessive irradiation can cause significant damage to cellulose. It remains unclear whether such damage is caused by the light itself, the oxidizing medium, pH conditions, or the interaction of these factors.

Therefore, this study focuses on applying a 2^3 factorial experimental design to determine whether the influencing factors act independently or in combination to achieve pulp brightness and delignification, and to assess the potential consequences of these oxidative media on fiber integrity under photooxidative conditions.

Method

For good data reproducibility, it is necessary to select and control the required variables in an experiment. In the case study of cellulose and paper, a single type of wood must be chosen as the raw material, which is treated using the Kraft method. The selected wood is from the species "Pine," which has long fibers and a known lignin content, making it suitable for treatment in the digester and during the bleaching steps. The species *Pinus pseudostrobus* Lindl, also known as "White Pine," "Ortiguillo Pine," "Canis Pine," or "Pasingo Pine," was obtained from the municipality of Nuevo San Juan Parangaricutiro, Michoacán, Mexico.

After clearly determining which type of wood will be used, the following steps must be followed:

1. Screening of the wood with a thickness of 3 to 7 mm.
2. Kraft digestion of the wood.
3. First bleaching with chlorine dioxide.
4. Advanced oxidation process: photolysis of peroxide based on a design of experiments.

The focus of the present research is on the final stage of the process, while steps 1 to 3 correspond to the production of semi-bleached pulp—a method currently employed in the industry. Based on the experience reported by other authors, incorporating an acid treatment step is recommended to remove components such as hexenuronic acid, which helps prevent color reversion (An 1998). Additionally, no sensitizers were used in the medium to enhance light absorption, as reported by Machado et al. (1996) and Castellan et al. (1999).

Design 2^3

Based on the 2^3 design, this approach allows for the estimation of main factor effects with the same precision as varying one factor across all experiments. It minimizes confounding and maximize accuracy, providing the independent variables in a hierarchical order (Anup and Saikat 2018). A single type of wood will be used, along with other considerations to eliminate variability in the experiment and a confidence level of 95%. Additionally, pretreatments will be applied.

The experimental design was based on results reported by various authors (Machado et al. 1996, Perez et al. 1998, Castellan et al. 1999, Felício et al. 2003, Ouchi et al. 2003, Ouchi[1] 2008, Ouchi[2] et al. 2008, Eren 2018), demonstrating that alkaline media yield the best outcomes for cellulose bleaching when combined with ultraviolet light and the generation of perhydroxyl radicals via hydrogen peroxide. The variables of interest are presented in Table 1. The experiment was conducted with three replicates.

Table 1
High and low levels for each factor

Variable	Experimental Range
H_2O_2	1–2%
pH	2.18–11.15
Irradiation time	0-120 min

Temperature has been reported as a significant factor in the activation of peroxide during pulp bleaching; however, in this study a constant temperature was used (Castellan et al. 1999). Residence time shows a wide range of operating values depending on the type of irradiation source used, varying from 20 to 210 minutes (Machado et al. 1996; Perez et al. 1998; Ouchi et al. 2001, Ouchi[2] et al. 2003, Ouchi[1] 2008). Moreover, light effectiveness is more related to the number of photons absorbed than to residence time (Kurosu et al. 2009). In this study, the irradiation time was kept constant. Based on these considerations, the experimental design is shown in Table 2.

Table 2
Experimental Design 23

Treatment	pH	H_2O_2	UVC light
1	3	1%	with
a	11	1%	with
b	3	2%	with
ab	11	2%	with
c	3	1%	without
ac	11	1%	without
bc	3	2%	without
abc	11	2%	without

The experimentation was carried out in a batch-type photochemical reactor Fig. 1 designed by M.C. Gabriel Martínez and collaborators, which was modified to include a mixing system for the pulp. A high-pressure mercury lamp was used, whose emission spectrum is dominated by ultraviolet radiation, particularly at a wavelength of 253.7 nm, with a power output of 250 W.

For the characterization of bleached and unbleached cellulose, well-established methods are available to evaluate the physical and mechanical properties before and after processing as a cellulosic material used in papermaking. This research will be based on TAPPI standards. Table 3 shows the tests that will be analyzed.

Table 3
TAPPI standards

Standard	Analysis name
T 205 om-88	Standard Sheet Formation
T 221 om-93	Drainage Time
T 230 om-82	Viscosity
T 233 cm-82	Fiber Classification
T 236 cm-85	Kappa Number
T 402 om-93	Sheet Conditioning
T 404 cm-92/ISO 1924	Breaking Length
T 410 om-93	Basis Weight
T 411 om-89	Thickness
T 412 om-94	Moisture Content
T 414 om-88/ISO 1974	Tear Index
T 425 om-91	Opacity
T 460 om-88/ISO 3687	Gurley Porosity
T 525 om-92	Whiteness

Results and discussion

The following section presents the different analyses performed on the pulp before and after the oxidative treatment with ultraviolet light, aiming to identify variables that show significant changes and to determine whether the process influences pulp bleaching and its physicochemical properties. The experimental design for each treatment has already been shown. Table 4 shows the values that were used to start all the treatments.

Table 4
Different kappa numbers for the process steps

Step	Kappa number	Residual lignin (%)
Digester outlet	17.10	2.51
Exit from the chlorine dioxide bleaching stage	3.16	0.47

Table 5 indicates that, under the experimental conditions evaluated, the application of ultraviolet (UV) light by itself does not exert a statistically significant influence on the viscosity of Kraft pulp, nor on its main physicomechanical properties such as tensile strength, tear resistance, or burst index. This suggests that the structural integrity of the cellulose fibers remains practically unaltered by exposure to UV light alone in these conditions. On the other hand, the results demonstrate that both brightness and the degree of delignification are parameters that are sensitive to the presence of UV light, particularly when this irradiation is combined with alkaline pH conditions (Machado et al. 1996, Perez et al. 1998, Castellan et al. 1999, Felício et al. 2003, Ouchi et al. 2003, Ouchi[1] 2008, Ouchi[2] et al. 2008, Eren 2018). This behavior can be attributed to the photolytic activation of lignin chromophores and their enhanced solubilization in alkaline media, which favors partial removal or modification of residual lignin (Tylli 1996). Finally, the opacity values reveal an additional effect: when UV light is applied together with the three factors under study, a synergistic interaction occurs that modifies this property. This finding highlights that opacity is not only dependent on the inherent fiber structure but is also strongly influenced by photochemical reactions induced by UV radiation in conjunction with the chemical environment.

Table 5
Significance results for each factor and their combinations in each test

Treatments	Kappa	Viscosity	Brightness	Opacity	Physicomechanical tests
pH			√		
H_2O_2			√	√	
UV	√		√		
pH-H_2O_2					
pH-UV			√		
H_2O_2-UV					
H_2O_2-UV-pH				√	

The kappa number indirectly indicates the amount of residual lignin surrounding the fiber. An empirical reference suggests that dividing the kappa number by seven gives the approximate percentage of residual lignin relative to the cellulose content. In the initial and final values reported by Machado et al. (1996), an 87% remission was achieved from the initial value to the value obtained after ultraviolet light treatment. In the present study, a remission of 51.3% was obtained under the given treatment conditions, without losing viscosity. Kurosu et al. (2009) found that a reduction in the kappa number is strongly correlated with increased brightness when treated with UV light. Castellan et al. (1999) also succeeded in enhancing lignin degradation, obtaining lower kappa numbers in treatments using light at 300 nm.

The obtained data showed lower values where ultraviolet light was present compared to those without light exposure. This can be attributed to the delignifying effect of UV light in combination with peroxide, as reported by various authors (Machado et al. 1996; Perez et al. 1998; Ouchi et al. Ouchi et al.2001, Ouchi et al.2003, Ouchi[1] 2008). However, the interaction with other factors could not be determined until statistical analysis was performed. Once conducted, the analysis revealed that the UVC factor was the only statistically significant variable, with no interaction effects contributing to delignification. In Table 6, it can be seen that, for factor P, the only one with values of zero is the ultraviolet light factor, and in factor F, it is the one that exceeds the negative value, indicating its significance when applying a 95% confidence level.

Table 6
Coded coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		1.9792	0.0605	32.69	0.000	
pH	-0.0935	-0.0467	0.0605	-0.77	0.451	1.00
H2O2	0.0168	0.0084	0.0605	0.14	0.891	1.00
UV	-0.6330	-0.3165	0.0605	-5.23	0.000	1.00
pH*H2O2	-0.0065	-0.0032	0.0605	-0.05	0.958	1.00
pH*UV	-0.0343	-0.0171	0.0605	-0.28	0.781	1.00
H2O2*UV	0.0427	0.0214	0.0605	0.35	0.729	1.00
pH*H2O2*UV	-0.0077	-0.0039	0.0605	-0.06	0.950	1.00

Viscosity provides an indirect measure of the degree of polymerization of the cellulose chain, which reflects the extent of degradation after the oxidative digestion and bleaching treatments. The results showed significant differences in viscosity among the samples. This finding was confirmed by the analysis of variance (ANOVA), which revealed statistically significant effects and/or interactions ($p < 0.00001$). The very small p-value provides strong evidence against the null hypothesis, indicating that the observed variations in viscosity are unlikely to be due to random experimental error. Figure 2 suggests that the combination of pH, peroxide concentration, and ultraviolet light tends to influence the viscosity response; however, at a 0.05 confidence level, the effect could not be confirmed. It is possible that with a lower confidence threshold, these factors might still fail to show a statistically significant effect.

Several authors agree that a certain loss of viscosity is acceptable. Machado et al. (1996) reported a decrease in viscosity from 19 to 12 cP, while achieving higher viscosity values when using TiO_2 as a catalyst, assuming it acts as a protective layer for cellulose, with results ranging from 420 to 625 dm^3/kg (Castellan et al. 1999; Pérez et al. 1998). Tsuji et al. (2012) also observed a decrease in viscosity when using ultraviolet light; however, they attributed this effect to the overall treatment conditions. Ouchi[1] (2008) found a correlation between viscosity and the type of emitted light, reporting lower viscosity values at shorter wavelengths. Similarly, Hwang and Lucia (2004) established a correlation between viscosity, exposure time, and the degree of delignification.

In the present study, it is assumed that such changes were not observed due to the reactor configuration and irradiation conditions, specifically the lamp power, exposure time, and emitted wavelength.

Brightness indicates the degree of light reflection on the cellulose surface. In practical terms, it is expressed as a percentage of light reflected by the paper sample compared to that reflected by a standard reference material (a pellet of pure magnesium oxide). The reflected light is measured at a wavelength of 457 nm (Escoto 2004).

The results showed noticeable differences among treatments, particularly in response to pH. Treatments in acidic media exhibited a decrease in brightness, confirming previous findings reported by other authors, where alkaline conditions enhance the bleaching action on cellulose. Additionally, it was confirmed that both peroxide and ultraviolet light independently contribute to bleaching and can achieve this effect on their own. It was found that the interaction between ultraviolet light and pH is a significant factor to consider when optimizing the bleaching process, in contrast to what was expected, namely that light and peroxide would interact through the formation of the hydroxyl radical.

In Fig. 3, it can be observed that pH is the most significant factor in the treatment, followed by the interaction between ultraviolet light and pH. Once again, ultraviolet light is identified as a key factor influencing the response. In this case, brightness serves as an indicator showing that the combination of pH and UV light is effective in achieving higher brightness compared to conventional treatments. Finally, peroxide concentration also falls within the range of parameters that can contribute to increased brightness.

The opacity of paper is expressed as the difference between the light that passes through the sheet and the light that is reflected. In other words, the more light that passes through the paper, the more transparent or less opaque it is. Thus, opacity is defined as the property of paper that prevents visibility through it (i.e., transparency).

The analysis of variance (ANOVA) showed high p-values for most factors and their interactions, indicating no significant effects overall (Table 6). However, the peroxide concentration and the combination of all three factors exhibited values suggesting a notable influence on opacity (Fig. 4), deviating from the expected norm. This suggests that the combined effect of these factors, particularly peroxide, may alter the crystallinity of the cellulose sample. This could lead to the hypothesis that these conditions contribute to hemicellulose degradation.

The drainage time indicates the degree of refining of the pulp, referring to the fiber size after the mechanical refining treatment. It is expressed as the time it takes for a fluid (in this case, water) to pass through a mat of compressed and densely packed fibers. The shorter the fibers, the more difficult it is for the water to flow through the fiber network. These measurements are reported using the Canadian Standard Freeness (CSF) or the Shopper-Riegler index.

Breaking length is a measure of the mechanical strength of paper and represents the maximum length of a paper strip that can support its own weight without breaking when hung vertically. Higher values indicate stronger paper. The tensile index measures the bonding strength between fibers within the paper sheet. In this test, a strip 15 mm wide and 10 to 20 cm long (depending on whether the paper is formed in standard laboratory sheets or produced by an industrial paper machine) is subjected to a tensile force applied at one end until it breaks.

The data obtained showed a significant effect on the tensile index; however, this factor did not affect the breaking length response, and no statistical significance was found at a 0.05 confidence level.

Commonly referred to as the Mullen Burst Index, this test measures the bonding strength between fibers. Its primary purpose, both historically and currently, is to estimate the paper's resistance to bursting (rupture) during use (Escoto 2004) In this study, neither the obtained data nor the analysis of variance showed any significant changes, indicating that irradiation in an oxidizing environment does not affect these properties.

The Pareto chart illustrates the various factors and their interactions influencing the burst index response, showing that neither individual factors nor their combinations have a significant effect on this variable.

Tear resistance, measured by the Elmendorf test, is a classic method developed to specifically assess a particular property of paper. The test consists of applying a load (pendulum) perpendicular to the paper surface until complete rupture occurs (Escoto 2004). The energy or work required to tear the paper is measured in Newtons or gram-force. Similar to the burst index, neither the data nor the analysis of variance showed any significant changes in this test.

Porosity, similar to drainage time, reflects the arrangement of fibers and the pores formed between them when they are laid out in a paper sheet. It is measured by passing 100 cc of air through the sheet and recording the time it takes for this volume of air to pass through.

Folding endurance indicates the strength and cohesion of cellulose fibers in a paper sheet. It is measured by repeatedly folding the paper until it breaks; the number of folds is recorded, and the folding endurance is expressed as the base-10 logarithm of the total number of folds before failure.

Conclusions

The study demonstrates that ultraviolet (UV) light irradiation, when applied in an alkaline peroxide medium, effectively enhances the brightness and degree of delignification of Kraft pulp derived from *Pinus Pseudostrobus* lindl. Statistical analysis confirms that UV light significantly influences these bleaching parameters, particularly impacting brightness and the kappa number, which reflects residual lignin content. Importantly, the application of UV in this context does not adversely affect the viscosity or the physico-mechanical properties of the pulp, indicating that the treatment preserves its structural integrity. The findings suggest that integrating UV irradiation into the bleaching process is a promising strategy for pulp whitening, potentially offering a more environmentally friendly and energy-efficient alternative to conventional methods. Further research with a more detailed statistical approach and exploration of additional process variables such as temperature and light sensitizers could optimize this technique for industrial application.

Declarations

Funding Declaration

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Author Contribution

The contributions of the individuals involved in this study were distributed as follows:- Alfonso Vargas Santillán was responsible for conducting the experimentations and performing the statistical analyses.- José Guadalupe Rutiaga Quiñones provided valuable consultations, made substantial contributions regarding the topics of light and electromagnetic energy, and also contributed to the drafting and creation of text and context within the manuscript.- Gabriel Martínez Herrera served as the project's advisor and coordinator, overseeing the acquisition of materials, managing contacts, and ensuring the overall execution of the project.Additionally, Marco Antonio Martínez Cinco contributed by facilitating the necessary spaces and establishing the methodology for the equipment used in the measurements.

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Figures

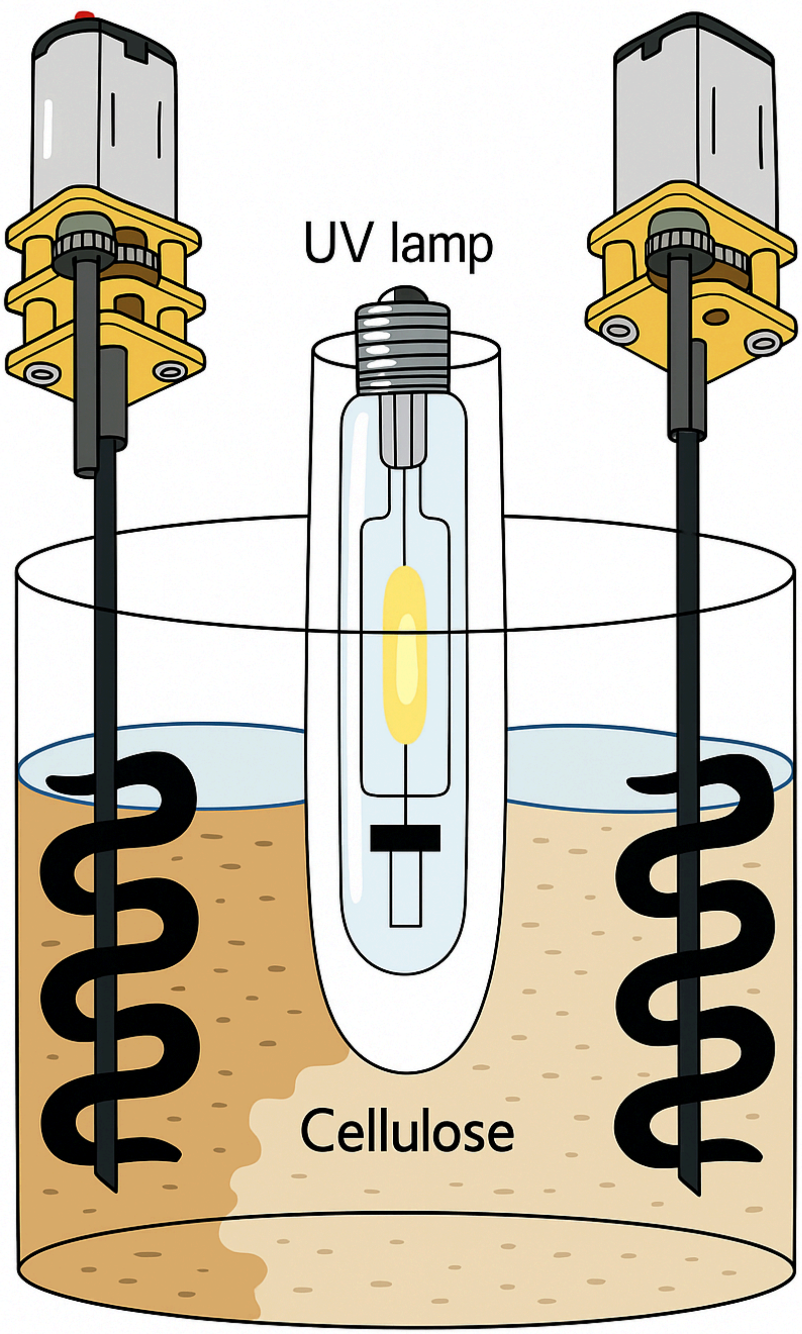

Figure 1

Reactor Diagram

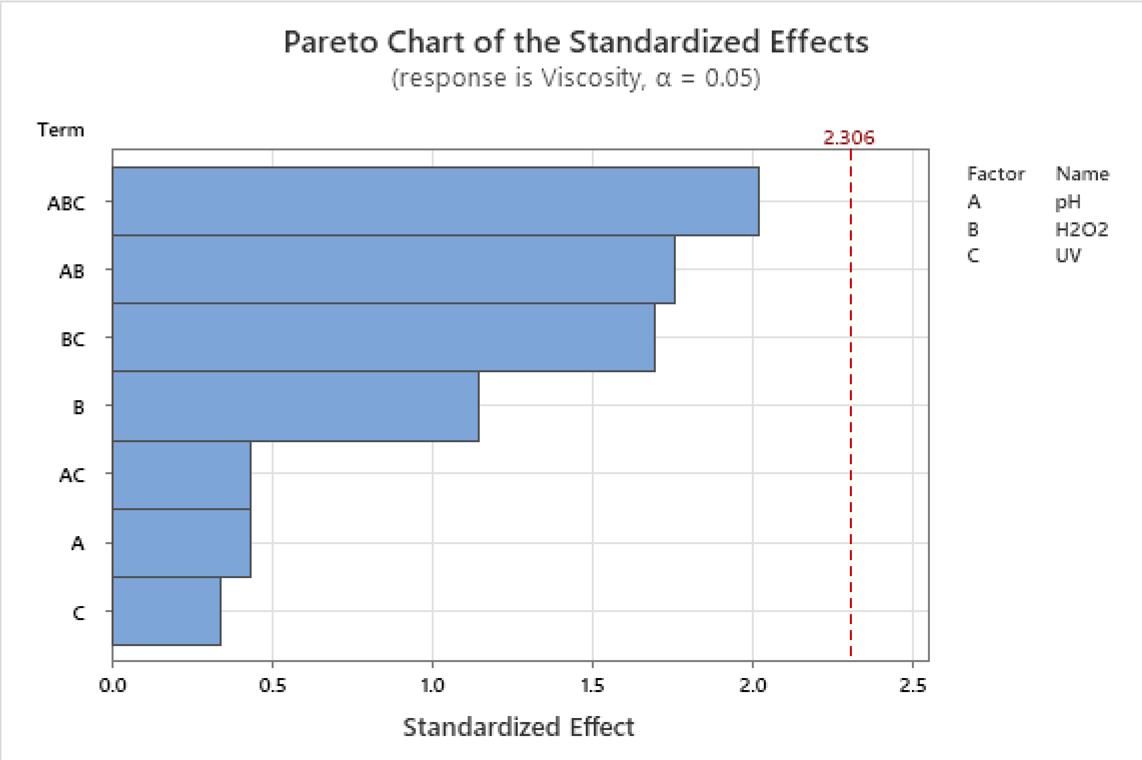

Figure 2

Significant values in viscosity

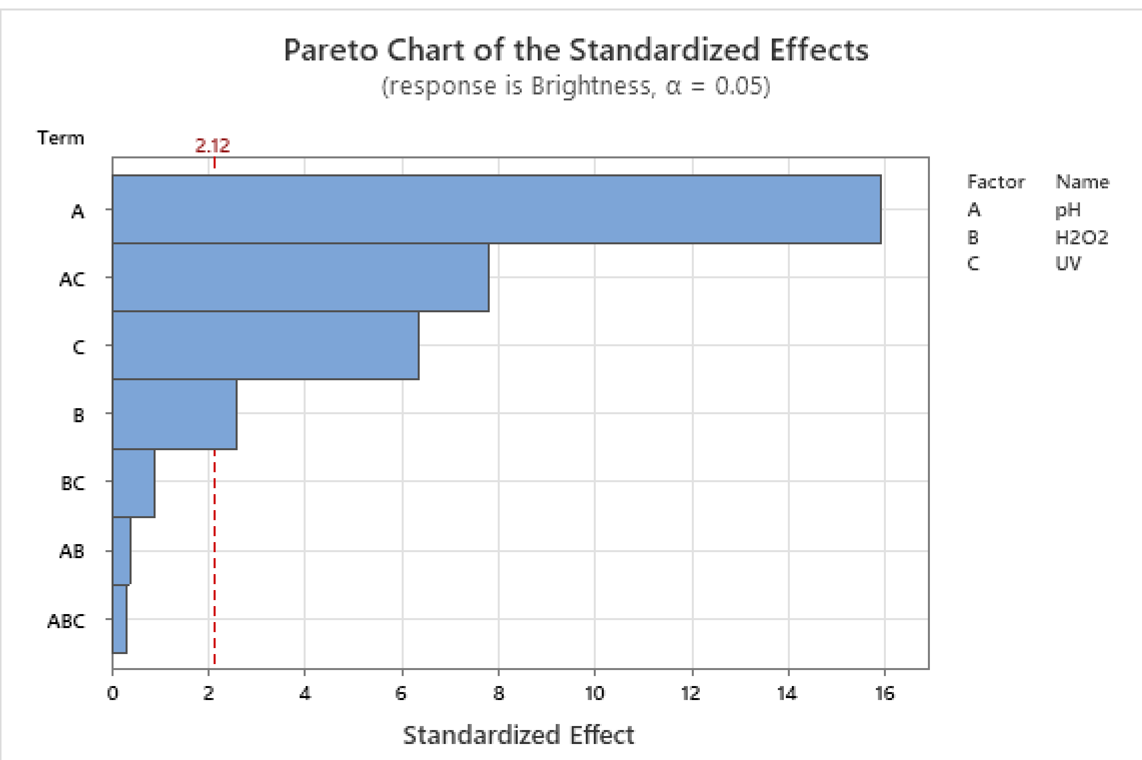

Figure 3

Significant values for brightness

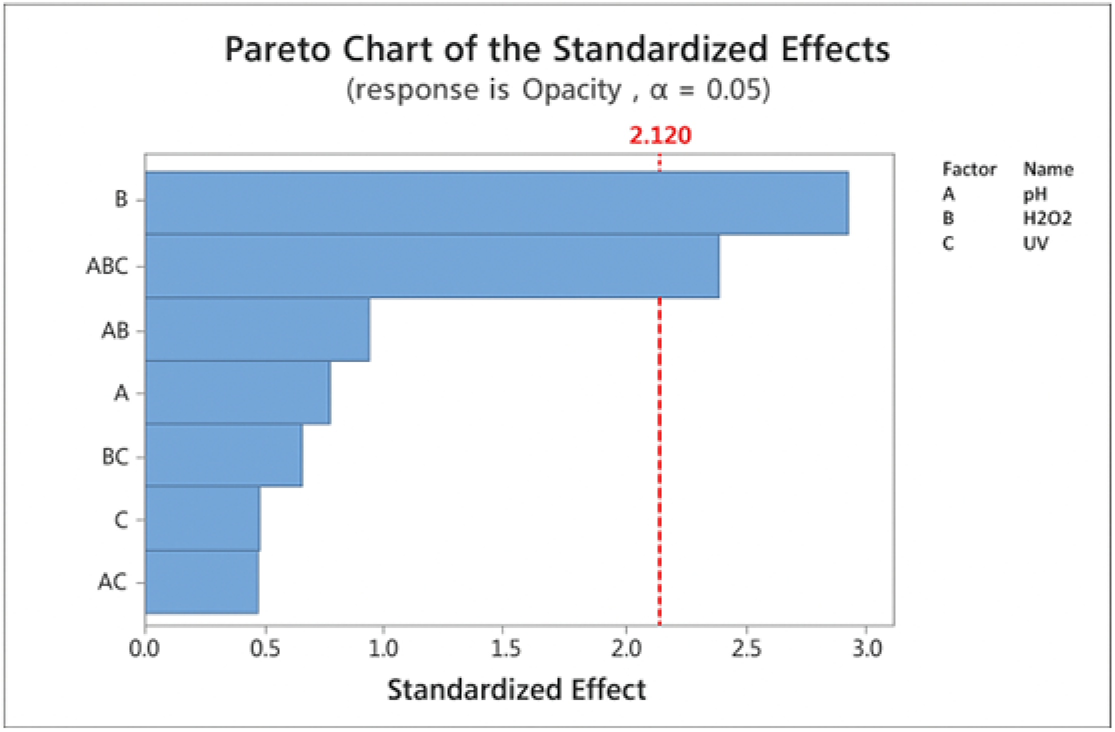

Figure 4

Significant values for Opacity