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TÍTULO DE LA TESIS

**ELABORACIÓN DE MATERIALES FOTOSENSIBLES
PARA REGISTRO DE REJILLAS HOLOGRÁFICAS**

Tesis para obtener el título de

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PRESENTA

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RESUMEN

Elaboración de materiales fotosensibles para la obtención de rejillas holográficas grabadas en tiempo real bajo condiciones ambientales de laboratorio, a partir de la caracterización del comportamiento de películas fotosensibles hechas con alcohol polivinílico, sales metálicas como dicromato de amonio, cloruro cúprico, un fotopolímero comercial a diversas concentraciones, un tinte comercial y un colorante aniónico.

Principalmente se realizó el siguiente análisis: las eficiencias de difracción con respecto a la energía y la fotosensibilidad de los materiales realizados.

Las películas fotosensibles de cloruro cúprico mezclado con un tinte sintético y alcohol polivinílico mostraron mayor eficiencia de difracción comparada con la mezcla de cloruro cúprico con alcohol polivinílico (PVA) sin embargo las películas fotosensibles compuestas de dicromato de amonio y la variación de hidrolisis del alcohol polivinílico, produjeron mejores resultados en su eficiencia de difracción, esto se debe a que el dicromato de amonio presenta mejores propiedades de fotosensibilidad. Por otro lado, al mezclar un fotopolímero líquido con un colorante aniónico este se vuelve sensible a la región visible lo que permite obtener rejillas holográficas entre la interferencia de dos longitudes de onda las cuales mostraron cambios en el índice de refracción.

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Con todo mi cariño y amor a mi familia EMKML ♡

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Este logro es más tuyo que mío. Estuviste conmigo 17 años de mi vida, dándome todo lo indispensable y sacrificando muchas cosas, incluso tu vida para que yo pudiera ser lo que soy, quizás te quedaste a la mitad de camino, pero lo que continua de él yo trataré de que sea como tú lo hubieras querido.

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INTRODUCCIÓN GENERAL

En 1947, antes de que se inventara el primer láser, Dennis Gabor (1900-1981), intentó mejorar la resolución y definición del microscopio electrónico mediante un registro fotográfico de imágenes al que llamó holograma, del griego *Holos*, que significa completo, y *Gramma* que significa imagen. Esta palabra se usa para definir toda la información colectada por el material fotosensible (holograma) donde se graba toda la información de amplitud y fase del objeto [1].

El método ideado para el proceso holográfico se compone de dos pasos: el primero en la amplitud y la fase en el registro del holograma, y el segundo es la reconstrucción.

El primer paso consiste en que el objeto se graba en un medio fotosensible, es decir, que se hace interferir el frente de ondas provenientes del haz del objeto con el frente de ondas del haz de referencia bajo condiciones de proceso de grabado como: coherencia, tiempo de exposición, eficiencia de difracción, etc.

Cuando la información es grabada en el medio fotosensible, esta es procesada para fijar la información, este medio constituye lo que se llama holograma que contiene un patrón de interferencia producido por la interacción coherente del haz de referencia y el haz de luz proveniente del objeto.

En el segundo paso, el holograma se ilumina con un frente de ondas, el cual, al difractarse en el medio fotosensible debido al patrón grabado en éste, una porción de la luz difractada reconstruirá el objeto. Si el haz de iluminación es similar al haz de referencia y está localizado en la misma posición que este, la imagen del objeto estará en la misma posición que la del objeto original, si el haz de reconstrucción es diferente se tendrá una variación en la imagen reconstruida, siendo amplificada o desamplificada, en diferente posición y con aberraciones [2].

En el registro holográfico, se usa un material fotosensible como una placa fotográfica donde se graba el patrón de interferencia, el cual es producido por la superposición de dos haces: el haz de luz reflejada o transmitida directa por el objeto, y el haz de luz dispersada por el objeto mediante reflexión o transmisión. La reconstrucción consiste en incidir un haz de luz luminoso a través del registro fotográfico (holograma), donde la luz al pasar se difracta de tal manera que toma la forma del

frente de onda original que propaga el objeto inicialmente iluminado, entonces el objeto es reconstruido [3]. Aunque Dennis Gabor, no tuvo éxito en mejorar el microscopio electrónico, encontró un método nuevo para formar imágenes y así las bases de la holografía fueron establecidas. Posteriormente con el desarrollo del láser (fuente de luz coherente) este método fue mejorado por los investigadores Leith y Uptnieks [4].

Las aplicaciones de la holografía son bastas y las de mayor desarrollo son: la proyección en 3D, la tecnología de elementos ópticos de difracción, el almacenamiento de la información, memorias holográficas, rejillas, sensores, hologramas de identificación y hologramas para presentación de tipo comercial (empaques, etiquetas, tarjetas de felicitación anuncios, juguetes, llaveros, etc. Los cuales se encuentran en constante evolución.

Actualmente existe una diversidad de literatura científica que describe a ciertos materiales como medios de registro holográfico (en general sales metálicas) y el desarrollo de éstos para una gran variedad de usos en la óptica holográfica; actualmente el campo de investigación de materiales para el registro en tiempo real o replicación holográfica es objeto de estudio en varios centros de investigación en óptica a nivel mundial.

De ahí la importancia de las investigaciones en el campo de los materiales de registro holográfico adecuados al avance y desarrollo de dicha área [5].

Se han desarrollado un gran número de materiales útiles para el registro holográfico, tales como fotopolímeros, foto-refractivos orgánicos e inorgánicos, gelatinas dicromatadas, gelatinas sensibilizadas de haluro de plata, fotorresinas, vidrios sol-gel, termoplásticos, fotocrómicos y fotodicroicos [6].

La gran cantidad de materiales disponibles se debe a la ausencia de un material de registro que posea todas las características requeridas para cada una de las aplicaciones, es decir, espesor suficiente, alta capacidad de modulación de índice de refracción, altos rendimientos en difracción, alta sensibilidad, buena calidad óptica con bajos niveles de dispersión de luz y pérdidas por absorción, estabilidad dimensional durante la grabación del holograma y su posterior utilización, buena estabilidad térmica y química [7].

La teoría y las técnicas experimentales reportadas para la obtención de un holograma son importantes, así como la teoría de los materiales, que juega un papel preponderante en el medio del registro donde el holograma es grabado.

El Laboratorio de Holografía y Materiales del Instituto Nacional de Astrofísica, Óptica y Electrónica (INAOE), México, ha reportado la investigación de materiales que representan nuevas alternativas para el registro holográfico, fundamentado principalmente en la fácil adquisición y el costo accesible; además, del mejoramiento de las propiedades fisicoquímicas y ópticas de los materiales ya conocidos como la gelatina dicromatada o el alcohol polivinílico.

La mayoría de las emulsiones utilizadas para construir las capas de película como medio de registro holográfico consisten básicamente en dos partes: el medio de suspensión (el cual debe satisfacer varios requisitos) y el compuesto sensible a la luz.

El presente estudio se enfocó en la implementación de nuevos materiales que puedan servir como agentes fotosensibilizadores en la generación de películas holográficas para su grabado en tiempo real y la obtención de rejillas de difracción para uso holográfico.

HIPÓTESIS

La elaboración de nuevos materiales para el registro holográfico a partir del dopado de polímeros tal como alcohol polivinílico, adhesivos con sales metálicas o colorantes tanto sintéticos como aniónicos, presentan una buena eficiencia de difracción, alta sensibilidad, durabilidad en almacenamiento y de bajo costo. Además, se espera que el comportamiento físico y químico de los materiales empleados tengan un mejor comportamiento y estabilidad en el registro holográfico, de rejillas de difracción.

OBJETIVO GENERAL

Preparar materiales para obtención de rejillas holográficas grabadas en tiempo real mediante la técnica de interferencia holográfica, empleando materiales como, colorantes sintéticos y aniónicos, sales metálicas y alcoholes (alcohol polivinílico).

OBJETIVOS PARTICULARES

Para cumplir con el objetivo general de esta tesis es necesario tener conocimiento de estos materiales con relación a sus características y propiedades que presentan, lo cual será posible mediante los siguientes objetivos particulares planteados:

- Obtener mezclas combinando materiales dichos en el objetivo general, obteniendo materiales fotosensibles.
- Cuantificar la resolución del material a través de la interferencia óptica.
- Cuantificar la eficiencia de difracción con respecto a la energía de las rejillas holográficas obtenidas por cada material.

JUSTIFICACIÓN

Existen una variedad específica de películas comerciales para el registro holográfico. Generalmente todas tienen la capacidad de registrar información de muy alta resolución para poder grabar el patrón de interferencia. Esta especialización las hace costosas y específicas para determinadas longitudes de onda.

El trabajo está dividido en cuatro capítulos, los capítulos están estructurados como sigue:

En el capítulo 1 se describe el registro de rejillas holográficas en tiempo real con adhesivo Norland 65® y eosina amarilla.

En el capítulo 2 se analiza y se describe el estudio de rejillas holográficas de dicromato de amonio variando la concentración de hidrolisis de PVA (alcohol polivinílico).

En el Capítulo 3 se describe el registro de rejillas holográficas con colorante sintético, en un sistema de PVA y cloruro cúprico, finalmente en el capítulo 4 se exponen las conclusiones generales obtenidas de las investigaciones de este proyecto de tesis.

CAPITULO 1

Registro de rejillas holográficas grabadas en tiempo real con adhesivo Norland 65[®] y eosina amarilla

RESUMEN

Registramos rejillas holográficas en tiempo real en un material líquido fotosensible. Este material es el adhesivo óptico llamado Norland (NOA 65[®]) mezclado con tinte de eosina amarilla. El Norland 65[®] es un fotopolímero líquido transparente e incoloro que se cura cuando es expuesto a luz ultravioleta (UV) al mezclarse con eosina amarilla se vuelve sensible a la región visible. Para registrar las rejillas se utilizó la interferencia entre dos diodos láser, obteniendo rejillas que mostraron cambios en el índice de refracción. Usamos dos longitudes de onda diferentes, para obtener el registro de rejillas para el uso de holografía.

Real-time holographic gratings recorded in Norland Optical Adhesive 65 and yellow eosin

ABSTRACT

We recorded real time holographic gratings in a photosensitive liquid material. This material is Norland Optical Adhesive (NOA 65[®]) mixed with yellow eosin dye. The NOA 65[®] is a clear, colorless, liquid photopolymer that cures when is exposed to ultraviolet light (UV) but when is mixed with yellow eosin becomes sensitive to visible region. To record the gratings we used the interference between two diode laser, obtaining gratings by refraction index changes.

We use two different wavelengths, for recording the gratings and its highest diffraction efficient was obtained when the grating was illuminated with a wavelength.

INTRODUCCIÓN

The search in photosensitive materials for holography with certain disables characteristics is bigger each day. Some of this characteristics are their energy sensitivity, high response to wavelengths range, response in real time, high resolution, fast chemical develop, and bigger information storage capability [8-9].

In this paper we propose a theoretical model to analyses the behavior of the photosensitive material composed by the mix of the Norland Optical Adhesive No. 65 and eosin yellow dye, which has been reported as photosensitive material where it is possible to storage holographic gratings. The proposed model is function of the power of the source employed to recording the gratings. The proposed model is function of the thickness film, power of the source employed to recording the gratings.

The Norland optical adhesive 65[®] is composed by mercapto-ester and due its transparency in the visible region of the electromagnetic spectrum is used as adhesive for the optical materials, safety windows, or plane and holographic displays. This material is polymerized by direct ultraviolet light exposition [10]. The typical properties of NOA 65[®] are shown in table 1, we show its spectral transmission in fig.1

The applications of this polymer is for putting lenses in metal mounts, bounding plastic to glass and cold blocking by means of the cured process [11-12].

Table 1. Typical Properties of NOA 65[®]

| | |
|-----------------------------------|--------------|
| Solids | 100% |
| Viscosity at 25° C | 1200 cps |
| Refractive Index of Cured Polymer | 1.524 |
| Elongation at Failure | 80% |
| Modulus of Elasticity (psi) | 20,000 |
| Tensile Strength (psi) | 1,500 |
| Hardness - Shore D | 50 |
| Temperature Range | -15 to 60° C |

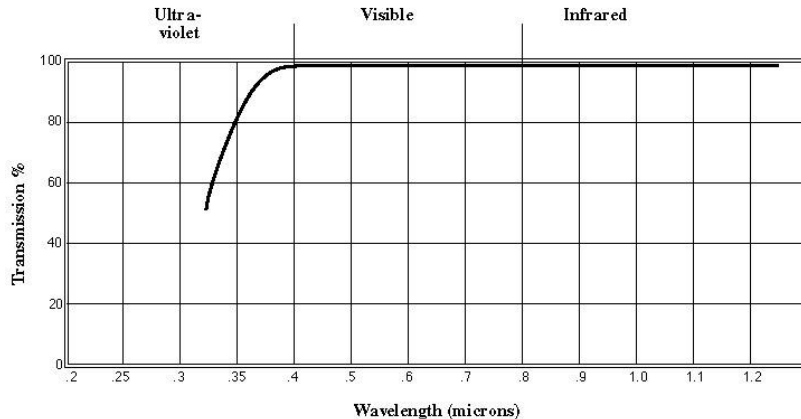


Fig. 1. Spectral transmission of NOA 65®

Yellow eosin is a pink water-soluble acid dye (anionic dye) which also displays yellow-green fluorescence and with wavelength of maximum absorbance (λ_{max}) of 517nm [13-14]. Yellow eosin is a xanthene dye and is used for the differential staining of connective tissue and cytoplasm. In histopathology, it is applied as a counterstain after hematoxylin and before methylene blue. It is also used as a background stain, thereby giving contrast to the nuclear stains. Its condensate formula is $C_{20}H_6Br_4Na_2O_5$, the chemical structure yellow eosin is presented in figure 2, [13].

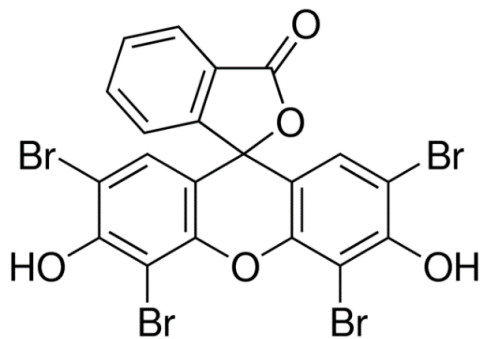


Fig. 2. Molecular structure yellow eosin

In this work we introduce the use of NOA 65® mixed with yellow eosin as photosensitive material, the sources used for the holographic recording was a diode laser 445-NL-500, at 420 nm and 532-NL-500, at 598 nm. With incident intensity on the photosensitive plate of 91.0 mW.

PREPARATION OF THE SAMPLE

The photosensitive material is prepared mixing (NOA 65[®]) and eosin yellow using a magnetic agitator. We prepare a concentration employing 0.15% of yellow eosin and 99.85% of NOA 65[®]. When the compounds are mixed, we deposited 0.012 gr into two coverslips. The mixture was introduced in the cell by the gravity technique as is shown in fig. 3.



Fig. 3. Photography of the deposition of the mix between two coverslips substrates by gravity technique.

Optical configuration was used with symmetrical arms of mirrors 1 and mirror 2, for the recording of holographic gratings on the photosensitive material we prepared. the source used for the holographic recording was a diode laser 445-NL-500, at 420 nm and 532-NL-500, at 598 nm. With incident intensity on the photosensitive plate of 91.0 mW. The main beam from the laser is divided into two arms using a cubic beam splitter (CBS).

A symmetrical angle is formed between both arms see figure 4 with 50% and 50% intensity in each arm. The two beams are overlapping on a point (interference zone) where is formed the diffraction grating.

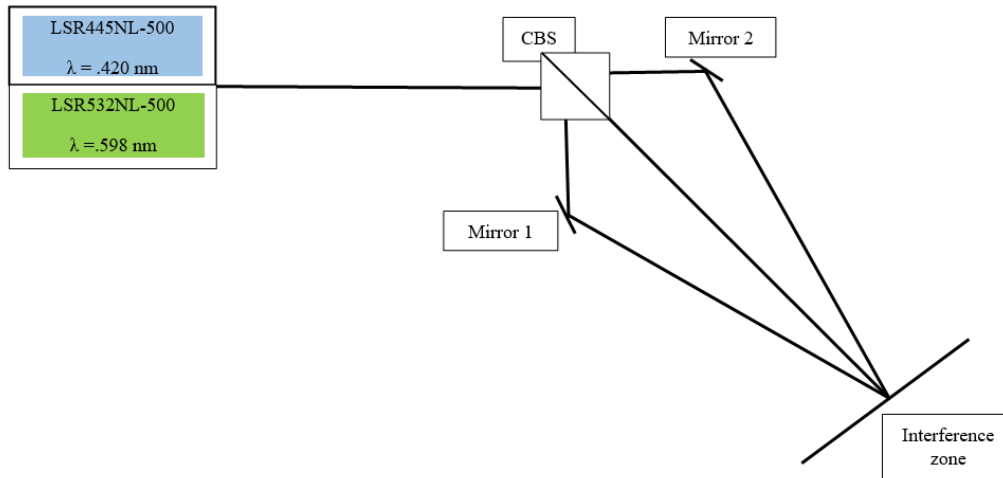


Fig. 4. Experimental setup with the optical components used to generate an interference zone and to record diffraction gratings.

RESULTS

We recorded a holographic grating on each emulsion, the exposure time for each emulsion was 9 minutes. When the emulsion begins to be exposed to the interference grating, we observe that the 1 and -1 diffraction orders appear, and its intensity increases as a function of time. We choose 9 minutes of exposure time because after this time, the diffraction efficiency stays constant.

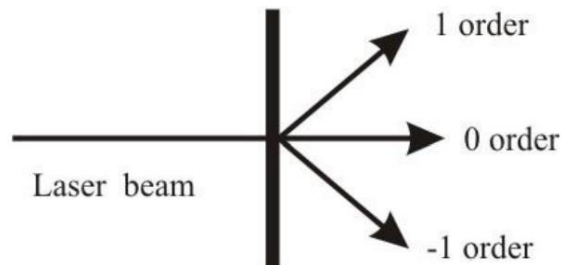


Fig. 5. Scheme of the diffracted order of the holographic grating. The diffraction efficiency is the ratio of the intensity laser incident beam and the 1 (or -1) diffracted order.

In Figure 6, we show a picture of the diffracted orders of the holographic grating made with diode laser 445-NL-500, with emission line at 420 nm when was illuminated by a red beam of a He-Ne laser.

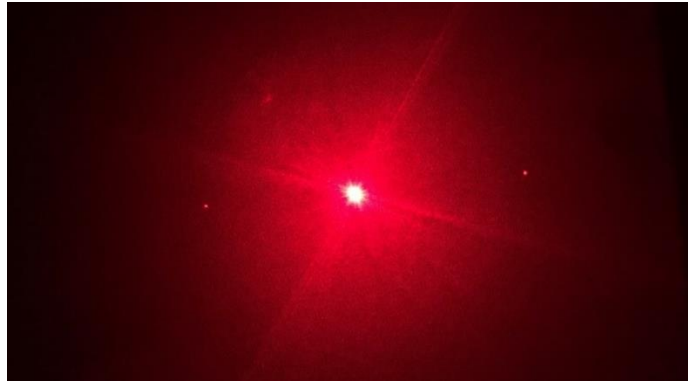


Figure 6. Diffraction pattern of the holographic grating recorded in the photosensitive film composed by NOA 65® and yellow eosin.

In Figure 7, we show a picture of the diffracted orders of the holographic grating made with diode laser 532-NL-500, with emission line at 598 nm, when was illuminated by a red beam of a He-Ne laser.



Figure 7. Diffraction pattern of the holographic grating recorded in the photosensitive film composed by NOA 65® and yellow eosin.

In fig. 8 we plot the intensity at first order of the photosensitive film using a diode laser 532-NL-500, with emission line at 598 nm.

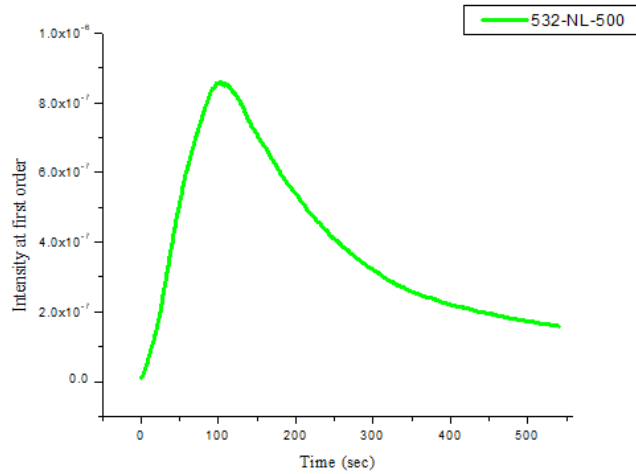


Fig. 8. Diffraction intensity at first order of the gratings composed by NOA 65[®] and eosin yellow.

In fig. 9 we plot the intensity at first order of the photosensitive film using a diode laser 445-NL-500, with emission line at 420 nm.

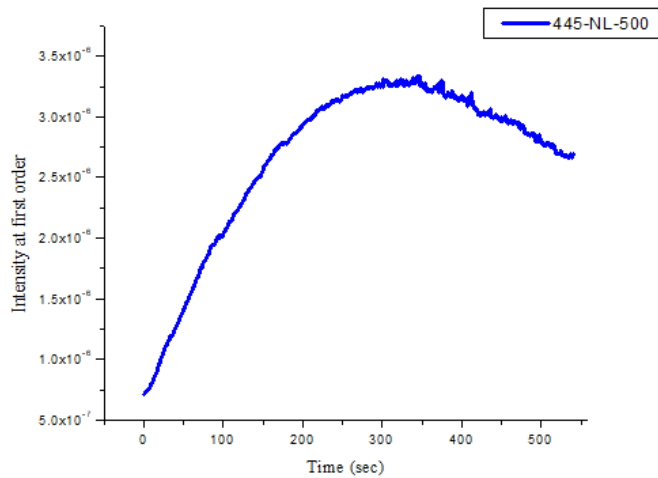


Fig. 9. Diffraction intensity at first order of the gratings composed by NOA 65[®] and eosin yellow.

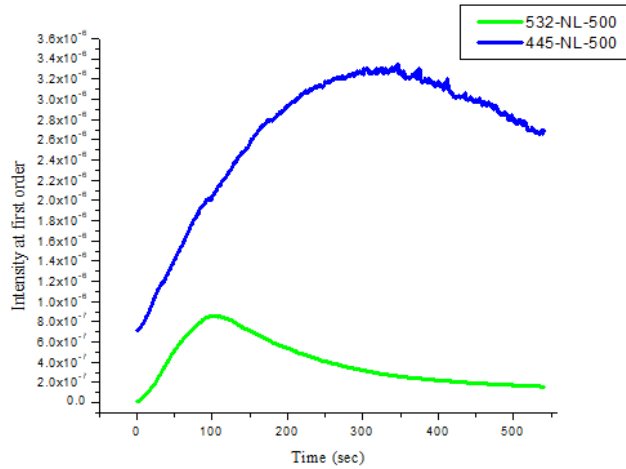


Fig. 10. Comparison of diffraction intensity at first order of the gratings composed by NOA 65® and eosin yellow using two diode laser 445-NL-500, with emission line at 420 nm and 532-NL-500, with emission line at 598 nm.

CONCLUSION

We show the use of Norland Optical Adhesive 65 mixed with eosin yellow as photosensitive material for the 420 nm and 598 nm. The best diffraction efficiency was obtained with the grating recorded using the 420 nm. This material proposed no requires chemical developing process.

CAPITULO 2

Estudio de rejillas holográficas de dicromato de amonio variando la concentración de hidrolisis de PVA (alcohol polivinílico)

RESUMEN

Mostramos el comportamiento de rejillas holográficas fotosensibles con la sal metálica dicromato de amonio $(\text{NH}_4)_2\text{CrO}_7$ a diferentes concentraciones de hidrolisis con PVA (alcohol polivinílico).

Las rejillas holográficas se registraron en tiempo real, observándose interesantes cambios en su eficiencia de difracción en función de la variación de hidrolisis de PVA.

Study of holographic gratings of ammonium dichromate varying the hydrolysis concentration of the PVA (poly (vinyl alcohol))

ABSTRACT

We show the behavior of the holographic gratings photosensitive with metallic salt, $(\text{NH}_4)_2\text{CrO}_7$ at different hydrolysis concentrations of PVA (poly (vinyl alcohol)). The holographic gratings were recorded in real time, observing interesting changes in their diffraction efficiency, as a function of the hydrolysis variation of PVA.

INTRODUCCION

In the characterization of materials it is used to work with diffraction gratings, due to the ease of measuring the diffraction efficiency of the point orders that were diffracted by the grating. A diffraction grating can be conceptualized as a set of elements transmitters and / or reflectors separated by a distance comparable to the wavelength of the light source in study [15]. This set of elements can be presented as a network of diffractive elements in the form of grooves or openings, or they can be grooves made in a given material [16].

Ammonium dichromate

It has been known since the early 1800s that the addition of water-soluble dichromate to certain biological organic colloids cause these materials to become photosensitive [17].

Ammonium dichromate $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$; has a molecular weight of 252.07, where Cr 41.26%, H 3.20%, N 11.11%, O 44.43%. This is prepared from ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$ and sodium dichromate $\text{Na}_2\text{Cr}_2\text{O}_7$ or by the interaction of ammonia gas and chromic acid in solution. $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$ as an appearance bright orange-red crystal, it is flammable, odorless and non-hygroscopic. Its structural crystal system is type monoclinic. It decomposes at about 180°C . The decomposition becomes self-sustaining at about 225°C with a spectacular swelling and evolution of heat and nitrogen, leaving Cr_2O_3 . Its heat of solution is 23.0 cal./g and it is very soluble in water. Solubility in water (w/w): 15.16% (0°C); 26.67% (20°C); 36.99% (40°C); 46.14% (60°C); 54.20% (80°C) 68.89% (100°C). Acid reaction: A 1% solution has a pH of 3.95 and a 10% solution has a pH of 3.45 [18]. The molecular structure detail of ammonium dichromate is shown in Figure 1.

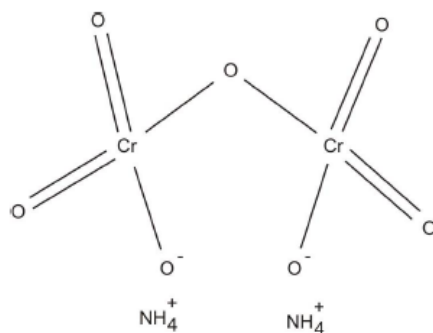


Fig. 1. Molecular structure of ammonium dichromate $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$

Polyvinyl alcohol

Polyvinyl alcohol (PVA) has relatively simple chemical structure with a pendant hydroxyl group; it is produced commercially from polyvinyl acetate, usually by a continuous process. The acetate group is hydrolyzed by ester interchange with methanol in the presence of anhydrous sodium methylate or aqueous sodium hydroxide. The physical characteristics and its specific uses depend on the degree of polymerization and the degree of hydrolyzed. The degree of hydrolysis is determined by the time point at which the saponification reaction is stopped.

Polyvinyl alcohol (PVA) is a synthetic polymer. It is presented in the form of granules or white powder [19].

Hydrolysis degree, or the content of acetate groups in the polymer, has an overall effect on its chemical properties and solubility. It affects the solubility of PVA in water [19]. The molecular structure detail of polyvinyl alcohol (PVA) is shown in Figure 2.

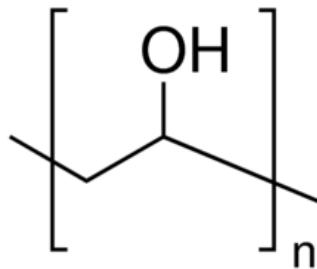


Fig. 2. Molecular structure of polyvinyl alcohol (PVA)

BACKGROUND

Holography is of major interest because of its potential applications in many fields from optical storage to art including medical imaging, structure in atomic scale. Among all the investigated systems, a large number of work has been devoted to dichromated materials such as dichromated -gelatin (DCG), -polyvinyl alcohol (DCPVA) and -polyacrylic acid (DCPAA) [20].

The photochemical behavior of dichromated polyvinylalcohol (DCPVA) films was analyzed upon exposure at 445 nm in connection with the hologram quality recorded in such a photosensitive material. The evolution of both involved species, chromium and polyvinylalcohol, were quantified by diffraction efficiency parameter of gratings made [20].

EXPERIMENTAL METHOD

To determine the best hydrolysis concentrations of PVA (poly (vinyl alcohol) at 10%, 15%, 25% with same quantity of $(\text{NH}_4)_2\text{CrO}_7$, we used three samples were analyzed.

Table 1. Concentration table

| Sample 1 | Sample 2 | Sample 3 |
|--|--|--|
| 2 gr of PVA 10% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20% | 2 gr of PVA 15% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20% | 2 gr of PVA 25% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20% |

PREPARATION OF THE FILM

- Area of substrate glass plates were 16 cm², used as substrate.
- On the glass substrate plates were poured 0.5 milliliters of the prepared mixture (each sample separated) and spread on the surface with the help of a dropper.
- The substrate with photosensitive film is set upon the spinner system for 2 seconds, applying 800 rpm to achieve the most uniform film possible.
- Storage the film at room temperature.
- Expose the sample

EXPERIMENTAL SETUP

Optical configuration was used with symmetrical arms of mirrors 1 and mirror 2, for the recording of holographic gratings on the photosensitive material we prepared.

The source used for the holographic recording was a diode laser 445NL-500, at 445 nm, with incident intensity on the photosensitive plate of 91.0 mW.

The main beam from the laser is divided into two arms using a cubic beam splitter (CBS).

A symmetrical angle is formed between both arms see figure 4 with 50% and 50% intensity in each arm. The two beams are overlapping on a point (interference zone) where is formed the diffraction grating.

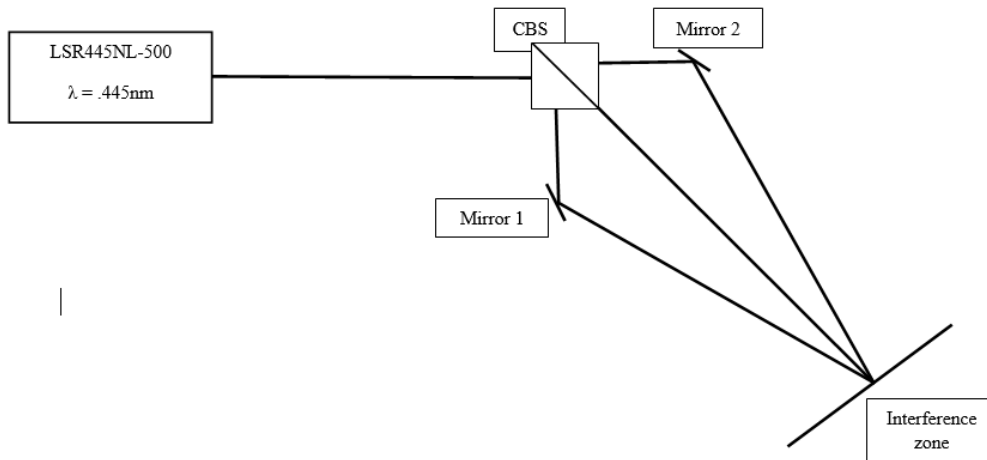


Fig. 3. Experimental setup with the optical components used to generate an interference pattern, to record diffraction gratings of Samples 1, 2 and 3 using a wavelength of 445 nm.

EXPERIMENTAL RESULTS

Diffraction behavior obtained from the photosensitive material prepared with 2 gr of PVA 10% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ at 20% (water and DC) (Sample 1) we show in figure (Fig. 4) the variations of the light intensity diffracted at first order with respect to exposure time.

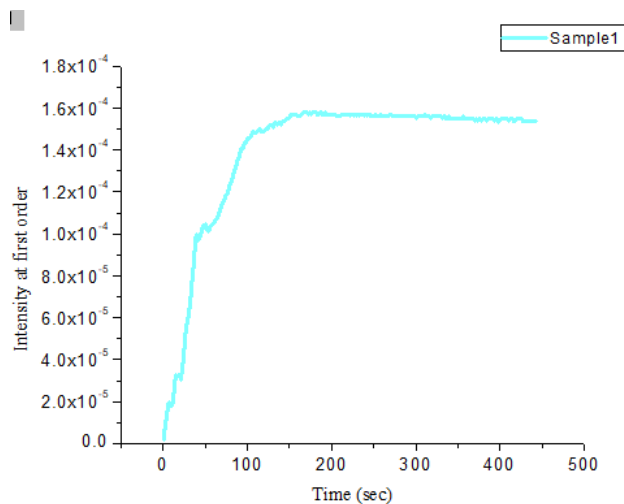


Fig. 4. Diffraction intensity at first order of the gratings made with sample 1 (2 gr of PVA 10% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20%)

Diffraction behavior from the photosensitive material prepared with 2 gr of PVA 15% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20% (Sample 2) we show in figure (Fig. 5) we could observe the variations of the light intensity, diffracted at first order with respect to exposure time.

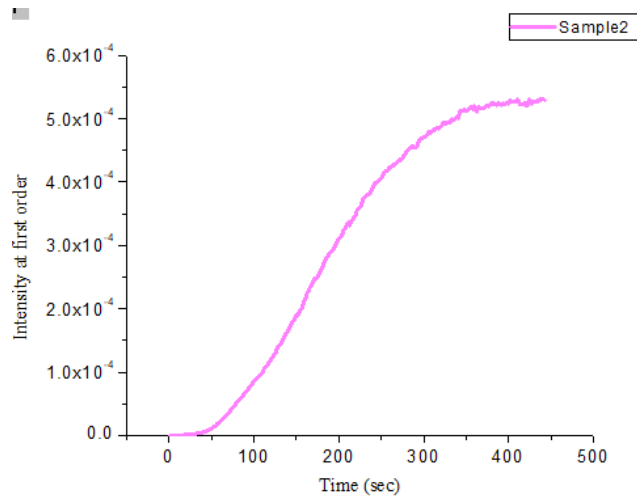


Fig. 5. Diffraction intensity at first order of the gratings made with sample 2 (2 gr of PVA 15% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20%)

Photosensitive material show the diffraction behavior, that were prepared with 2 gr of PVA 25% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20% (Sample 3) we show in figure (Fig. 6) Variations of the light intensity, diffracted at first order with respect to exposure time was observed.

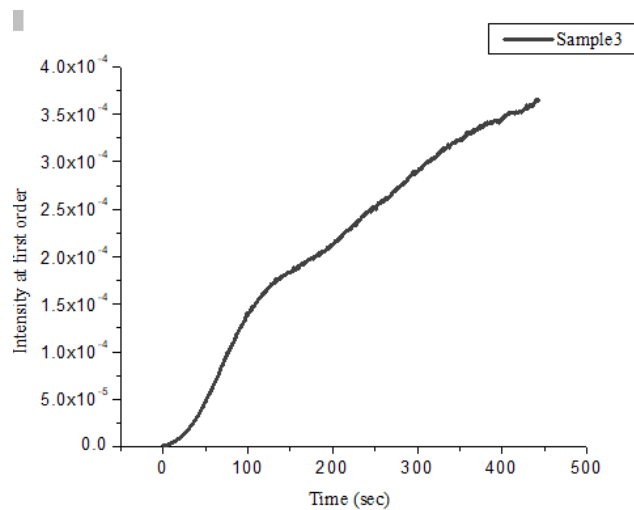


Fig. 6. Diffraction intensity at first order of the gratings made with sample 3 (2 gr of PVA 25% + 2 gr $(\text{NH}_4)_2\text{CrO}_7$ 20%)

The diffraction efficiency is one of the main parameters to determine if a type of compound is suitable for use in hologram recordings.

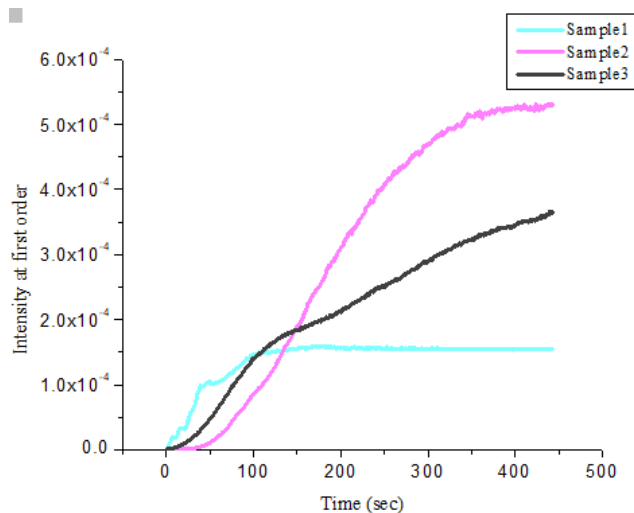


Fig. 7. Variations of intensity diffracted at first order, vs exposure time, for the variation in the concentration of sample 1, 2 and 3 is shown.

Figure 7 shows the behavior of the diffracted intensity distributions in the first order of the gratings comparing their behavior as hydrolysis function. The samples show three different responses due the hydrolysis of PVA material. Where it is observed that the sample 2 was the one that behaves better with respect to the diffraction efficiency but on the other hand with respect to the exposure time behaves better sample 1. As is showed in the figure.

CONCLUSION

Hydrolysis control is important parameter to design photosensitive materials, The PVA material has these characteristics, and it can be handled to obtain better results. As example, the fastest record was for gratings when PVA were added at 10% hydrolysis. However, the high efficiency was when PVA were added at 15% hydrolysis.

CAPITULO 3

Registro de rejillas holográficas con colorante sintético, en un sistema de PVA y cloruro cúprico

RESUMEN

Se presenta el análisis comparativo entre rejillas registradas hechas con cloruro cúprico y rejillas con una mezcla de cloruro cúprico y un colorante sintético. Se muestran resultados preliminares de las rejillas de difracción holográficas registradas, en las que el alcohol polivinílico (PVA) se mezcla con un agente fotosensibilizante, con la combinación de cloruro cúprico con colorante sintético. Las rejillas se registraron utilizando un diodo láser a una longitud de onda de 445nm. Los resultados experimentales de las rejillas registradas con cloruro cúprico el colorante sintético y PVA mostraron mayor eficiencia de difracción que las registradas compuestas únicamente de cloruro cúprico y PVA.

Holographic gratings recording in synthetic dye of system PVA and cupric chloride

ABSTRACT

Comparative analysis between gratings registered in cupric chloride and cupric chloride films, with synthetic dye is presented.

Preliminary results of recorded holographic diffraction gratings are shown, in which the polyvinyl alcohol mixed with a photosensitizing agent, cupric chloride and synthetic dye. The gratings were recorded using laser diode at a wavelength of 445 nm. Experimental results of gratings registered with cupric chloride and synthetic dye showed higher diffraction efficiency than those registered in cupric chloride.

INTRODUCTION

Holography is a way to record the optical information coming from a specific object, either reflected or transmitted by it, and this information is superposed with a reference beam, which is illuminated with a same laser light source.

New holographic recording materials it has been found that some synthetic dyes used for food show acceptable parameters of diffraction efficiency for holograms response; this was based using the McCormick® synthetic red dye.

A dye is a substance used as an additive in foods to recover its color, and to accentuate the original color or to give it a more attractive color. Among artificial or synthetic colorants, azo and non-azo dyes are distinguished. The former owes their color to the azo group $-N=N-$ conjugated with aromatic rings at both ends [21].

Table 1 Synthetic azo and non-azo dyes

| SYNTHETIC AZO-DYES | |
|-------------------------|---------------------------|
| Tartrazine (E102) | Red Allura AC (E129) |
| Orange Yellow S (E110) | Brilliant black (E151) |
| Azorubine (E122) | Brown FK (E154) |
| Amaranth (E123) | Brown HT (E155) |
| Red Ponceau 4R (E124) | Lithol Rubine BK (E180) |
| Red 2G (E128) | |
| SYNTHETIC NON-AZO DYES | |
| Quinoline Yellow (E104) | Indigo Carmine (E132) |
| Erythrosine | Brilliant Blue FCF (E133) |
| Patent Blue V (E131) | Acid Green BS (E142) |

BACKGROUND

In 1868 Witt postulates that all substances are organic compounds that contain in their structure functional groups that confer color, called chromophores, which contain valence electrons with relatively low energies of excitation, example of them are the functional group: azo ($-N=N-$); carbonyl ($C=O$) and benzene ($-C_6H_6$), among others. Within the same molecule there is another group of atoms that do not absorb radiation, but have the ability to modify the absorption of the chromophore, as well

as increase its intensity, this group was called autochromes. Examples of these are: oxydryls (-OH,) and amino (-NH₂), among others [22].

According to the FDA (Food and Drug Administration of the United States) a dye or pigment is any material that imparts color to another substance obtained by synthesis, extracted or derived, with or without intermediaries of the final change of identity, from a vegetable, animal, mineral or other source and that when added or applied is capable of impart color by itself [23].

ARTIFICIAL COLORANTS

Color is a property of matter directly related to the spectrum of light, so it can be measured physically in terms of radiant energy or intensity and its wavelength [24].

COLORS AS SENSITIZERS

In 1873 Vogel discovered that the addition of dyes to an emulsion makes it sensitive to the region where it absorbs the dye, thus a yellow dye sensitizes in the blue region and a blue dye sensitizes in the red zone [25].

In this research, dyes of artificial or synthetic origin were used. These were soluble in water, due to the presence of sulphonic acid groups (-SO₃), and consequently it easy to use, generally in the form of sodium salts, liquids and / or paste materials. They could also be used in insoluble form, such as lacquers with aluminum hydroxide, when they were added to solid products. In addition to being easier to use than natural dyes, artificial dyes were also, in general, more resistant to heat treatments, extreme pH, light, etc., than natural dyes [26].

A very common way to classify them is according to their chemical structure, according to the chromophores they contain and according to the compound from which they were derived, this are:

- Nitrous or nitro dyes
- Azo dyes or azo dyes
- Dyes derived from triphenylmethane
- anthraquinone dyes
- Indigo dyes

SYNTHETIC DYE COMPOUNDS

McCormick® red food colorants are a photosensitive agent; it is composed of two dyes mixed in water with glycol as a matrix preservative and propyl paraben. Characteristics of the dyes are that their molecules are composed of aromatic families (benzene), and 2 colors that are:

Allura Red AC

It is a synthetic red azo dye, it is a disodium salt and it has the appearance of a red powder, it is soluble in water and thermo-photosensitive. It is used as food dye and is listed as E129 (EU) or Red 40 (USA) codes; in most acids medium is stable as: citric, acetic, malic and tartaric acids. It becomes bluish in the presence of sodium bicarbonate at 10%, slightly bluish in the presence of sodium carbonate and ammonium hydroxide.

Characteristics:

1. **Formula:** $C_{18}H_{14}N_2Na_2O_8S_2$
2. **Molar Mass:** 496,42 g/mol
1. **IUPAC's Denomination:** Disodium 6-hydroxy-5-((2-methoxy-5-methyl-4-sulfophenyl) azo)-2-naphthalene
2. sulfonate
3. **Soluble:** Water. [26]

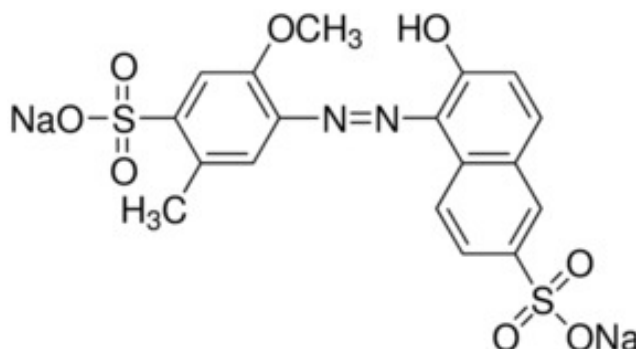


Fig. 1. Allura Red's molecular structure. [26]

Erythrosine:

It is also known as Red 3 (FDA) or E127; organ iodine compound used as violet red dye, for foods, printer's ink. It is soluble in water, glycerin, and propylene glycol, insoluble in ethanol. It is stable to heat, baking temperatures as well as being stable to light but is unstable in acid media. It can be chemically modified by incorporating

aluminum's structure to generate lacquers. The lacquers are insoluble in water and are widely used for their affinity for oily media.

Characteristics:

1. **Formula:** $C_{20}H_{14}Na_2O_5$
2. **Molar Mass:** 879,86 g/mol

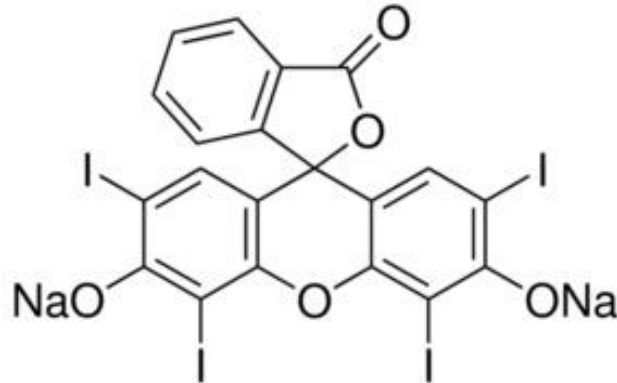


Fig. 2. Erythrosine molecular structure [26].

COMPOSITIONS OF THE FILM

Copper Chloride

Has the following properties:

- It is a solid blue-green dehydrate ($CuCl_2 \cdot 2H_2O$).
- Its relative density is 3.39 g / cm^3 .
- It has a molecular mass of 134.45 g / mol .
- Its melting point is $620 \text{ }^\circ \text{C}$.
- Its boiling point is $993 \text{ }^\circ \text{C}$.
- Copper chloride is soluble in water [27].

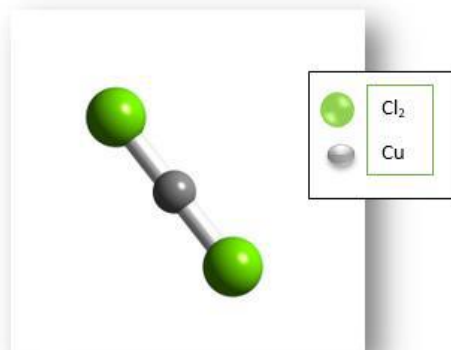


Fig. 3. Molecular structure of the $CuCl_2$ [27].

Polyvinyl Alcohol

Polyvinyl alcohol is produced commercially from polyvinyl acetate, generally a continuous process. The acetate groups are hydrolyzed by ester exchange with methanol in the presence of anhydrous sodium methylate or aqueous sodium hydroxide.

Physical characteristics specific functional uses depending on the degree of polymerization and the degree of hydrolysis.

Polyvinyl alcohol is classified into two classes: partially hydrolyzed and fully hydrolyzed [28].

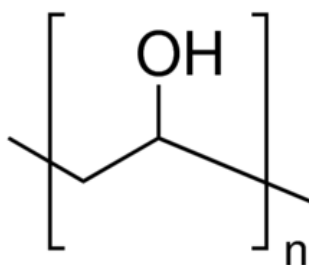


Fig. 4. Molecular structure of the Polyvinyl alcohol

EXPERIMENTAL METHOD

To determine the best concentration of McCormick® red dye, we were used three samples were analyzed, in which the concentrations of dye were changed; the cupric chloride and polyvinyl alcohol were with same concentration differentiated as follows:

Table 2. Concentration table: Sample B

| Sample 1 | Sample 2 | Sample 3 |
|--|--|--|
| 1.5% of dye + 1.6% of CuCl ₂ + 3 ml of PVA | 3% of dye + 1.6% de CuCl ₂ + 3 ml of PVA | 4.5% of dye + 1.6% de CuCl ₂ + 3 ml of PVA |

SAMPLE A

Cupric chloride + polyvinyl alcohol

To obtain the optimal work mixture, a sample was made which is composed of 1.6% cupric chloride and 3 ml of polyvinyl alcohol. This served to compare the three concentrations of samples B.

The mixing is carried out on a magnetic stirring for 25 minutes.

SAMPLE B

Cupric chloride + polyvinyl alcohol + synthetic dye

The three concentrations were performed as shown in table 2.

FILM GENERATION

1. Area of substrate glass plates were 16 cm², used as substrate.
2. On the glass substrate plates were poured 0.5 milliliters of the prepared mixture (samples A and B) and spread
 1. on the surface with the help of a dropper.
 2. It is carried to the spinner for 10 seconds applying 900 rpm to achieve a film as uniform as possible.
 3. Storage the film for 30 minutes at room temperature.
 4. Expose the sample.

SETUP INTERFEROMETRIC

Optical configuration was used with symmetrical arms of mirrors 1 and mirror 2, for the recording of holographic gratings on the photosensitive material we prepared.

The source used for the holographic recording was a diode laser 445NL-500, at 445 nm, with incident intensity on the photosensitive plate of 91.0 m W.

The main beam from the laser is divided into two arms using a cubic beam splitter (CBS).

A symmetrical angle is formed between both arms see figure 5 with 50% and 50% intensity in each arm. The two beams are overlapping on a point (interference zone) where is formed the diffraction grating.

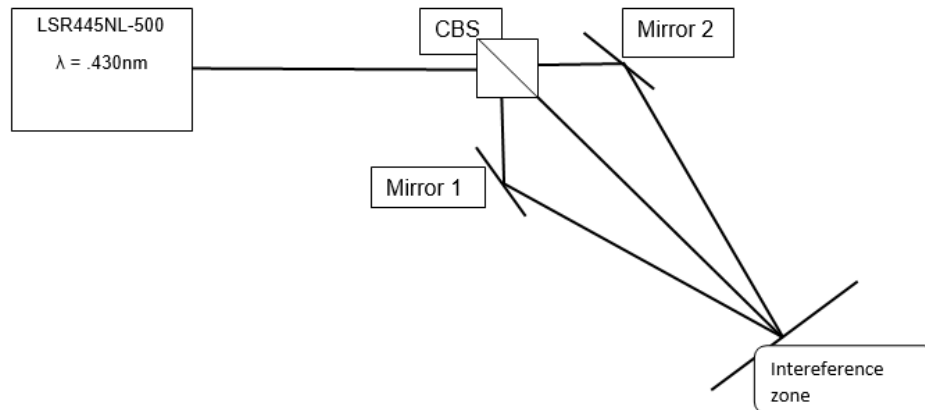


Fig. 5. Experimental setup with the optical components used to generate an interference zone and to record diffraction gratings of samples A and B, using a wavelength of 445 nm.

EXPERIMENTAL RESULTS

The diffraction behavior obtained from the photosensitive material prepared PVA+CuCl₂ (sample A), were showed in figure (6), we could observe the variations of the light intensity beam, diffracted at first order with respect to time.

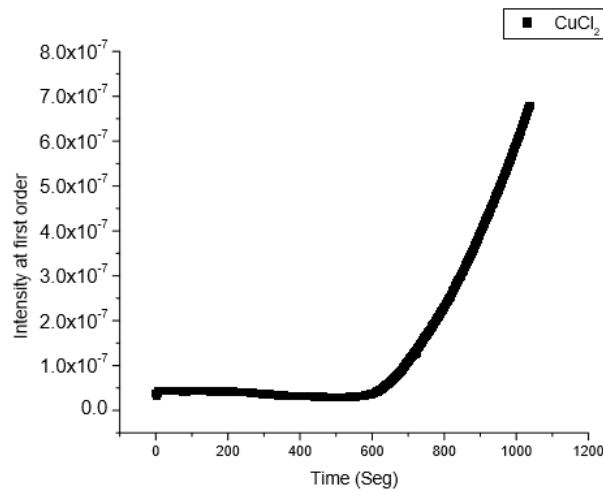


Fig. 6. Diffraction intensity at first order of the gratings made only with sample A.

The diffraction efficiency is one of the main parameters to determine if a type of compound is suitable for use in hologram recordings.

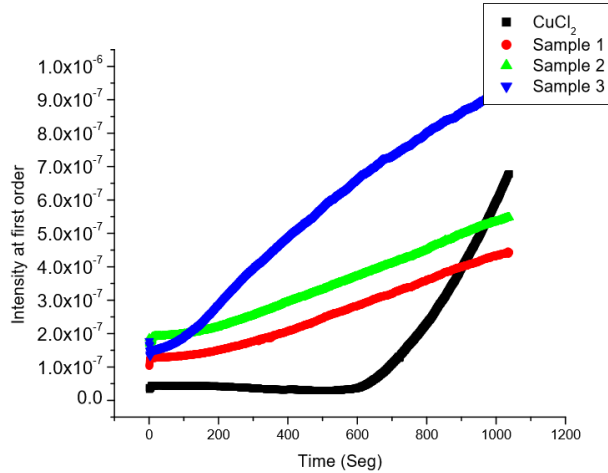


Fig. 7. The variation in the concentration of sample B, compared to sample A is shown.

Figure 7 shows the behavior of the intensity distributions diffracted in first order of the gratings comparing sample A with samples B; where it was observed that the sample 3 was the one that behaves better with respect to a faster time of engraving of gratings.

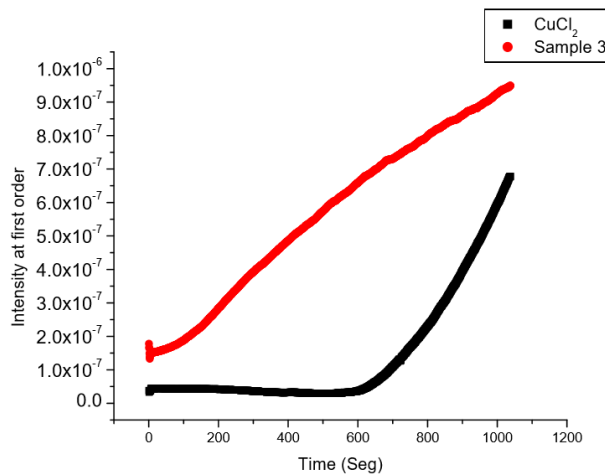


Fig. 8. Comparison of the sample A with sample 3.

Figure 8 shows the comparison of both samples, where the fastest recording was observed for gratings when synthetic dye was added.

CONCLUSION

From the results obtained, the fastest recording was observed for gratings when synthetic dye was added. And the register was more efficient, with the PVA + copper chloride + synthetic dye, with 4.5% concentration.

CAPITULO 4

CONCLUSIONES GENERALES

El estudio realizado en la presente tesis tuvo como objetivo general la obtención de rejillas holográficas grabadas en tiempo real mediante de la técnica de interferencia holográfica, empleando materiales como, colorantes sintéticos y aniónicos, sales metálicas y alcoholes (alcohol polivinílico). De los cuales se obtuvieron resultados favorables por cada capítulo, se realizaron mezclas para la obtención de rejillas holográficas combinando materiales que sirvieron para la cuantificación de eficiencia de difracción con respecto a la energía.

Se observó y cuantifico que las rejillas tienen diferentes comportamientos dependiendo del material del que están hechas y de la fuente de luz que se utilice.

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