

Wavelength Tunable Properties of Highly Oriented Colloidal Acrylate Latex Film

^aHelyati Abu Hassan Shaari, ^bMohammad Syuhaimi Ab. Rahman,
^aRahmah Mohamed

^a*Faculty of Applied Sciences, Universiti Teknologi Mara (UiTM),
Faculty of Engineering and Built Environment, Department of Electrical, Electronic and Systems
Engineering, Universiti Kebangsaan Malaysia (UKM)*

ABSTRACT

Colloidal acrylate latex film with wavelength tunable properties was simply fabricated by depositing colloidal monodisperse acrylate spheres with pre-vulcanized latex (PVL). The resultant film exhibited a reversible reflectance wavelength when highly oriented into high elongation upon interaction with incident light. Benefiting from the elastic characteristic of natural rubber latex, the film produced can withstand elongation up to 600% elongation to give various reflectance wavelengths at different strain levels. The reversible wavelength response of the colloidal acrylate latex film suggests its potential to be used as a main material for in situ monitoring of stress and strain devices.

Keywords: *wavelength tunable; strain; elastic-film*

1. Introduction

There has been rapid interest in the fabrication of colloidal film with wavelength tunable properties when highly oriented by means of stretching. Ordered spheres arranged along elastic film may diffract lights of selective wavelengths according to Bragg's Law (Schäfer et al., 2013). Any variation in distance between the spheres due to external stimuli such mechanical strain can lead to the change of the reflectance wavelength (Duan et al., 2011). Over the years, there are various kinds of photonic crystal with wavelength tunabilities being fabricated including gelled (Iwayama et al., 2003; Kanai et al., 2007), liquid (Schmidtke and Terentjev, 2010) and colloidal crystal (Fudouzi, 2009). The variety of photonic crystal can significantly expand their applications scope. Up to now, there have been number of demonstrations on wavelength tuning on the basis of colloidal crystals through various approaches. There are by varying sphere size (Zhang et al., 2011), magnetic field (Xia et al., 2012) refractive index contrast (Raman, Murthy and Hegde, 2011), angle of incident (Xuan and Ge, 2011), solvent (Wang and Han, 2011) and mechanical deformation (Sun, Yao and Gu, 2012). Yoshino et al. (1999) were the first to demonstrate the optical properties of elastic polymer opal prepared by polymer spheres. In their research, they reported that the diffraction lights can be controlled by the application of mechanical stress, proving its characteristics as mechanically tunable photonic material.

Most wavelength tunable film consists of two different materials, polymeric spheres and rubber. The choice of material that can be used as polymeric spheres is usually made from polymer colloids which later will form an orderly close packed structure. Since the spheres are rigid, elastomeric material such rubber is used to deformed the spheres by elongated the film to a certain extent (Li et al., 2004). Fudouzi and Sawada (2006) reported successfully synthesized colloidal monodisperse polystyrene (PS) spheres using emulsion polymerization method to produce periodically arranged spheres embedded in a silicone rubber. The wavelength tuning was obtained by reversibly changed the spheres distance with the deformation of the rubber matrix by stretching.

Here, this paper presents an alternative material that can be used for fabrication of colloidal film with wavelength tunable properties by varying stretch levels. The colloidal films reported in this paper consist of two parts, soft elastomeric part which is pre-vulcanized latex (PVL) and polymeric part which is acrylate spheres. Diffraction gratings are introduced to the sample film by creating order arrangement of acrylate spheres along the film to facilitate wavelength diffraction upon stretching.

crystals for biosensor applications such as filters, cavities and microlaser waveguides. On the engineering side, tunable photonic crystals could also serve as sensing materials to provide a platform for the in situ monitoring of mechanical strains by stretching or compressing. In strain-sensing applications, changes in the total refractive index by strain are caused by two effects: changes in the geometry of the holes in the crystal lattice and changes in the refractive index of the material (Tung et al., 2011). The ability of the stop band to shift and cover almost the entire wavelength of the visible region make tunable photonic crystal films a potential material for sensors, optical devices and display applications (Wang and Han, 2011).

Therefore, colloidal acrylate latex film with wavelength tunability properties being produced in this study may vary the applications of conventional rubber, leading likely to some new applications especially in optical engineering fields.

2. Experimental Methods

2.1. Materials

Full ammonia pre-vulcanized latex having low modulus 'LR Revultex' with 60.5 total solid content and 0.68 ammonia content was purchased from Revertex Sdn Bhd (Malaysia). Acrylate monomer, 99%, and potassium peroxy-disulfate (KPO), were purchased from Sigma-Aldrich Sdn Bhd (Malaysia) and used as received. Sodium stearate, was purchased from Fisher Scientific Sdn Bhd (Malaysia).

2.2. Synthesis of colloidal acrylate particles

The colloidal system in this study is an aqueous dispersion of acrylate particles which were synthesized using emulsion polymerization method. For a typical procedure, 106 ml acrylate monomer and 180 ml ion-exchanged double distilled water were added to a 1-L round bottom, 5-neck flask. The monomer was first heated at a temperature 60°C to remove the inhibitor. The initiator was potassium-peroxydisulfate (KPO), the emulsifier was sodium stearate and the medium was distilled water. The polymerization reactor consisted of a mechanical stirrer, a nitrogen gas inlet, and a thermometer to control the polymerization temperature. The acrylate monomer and distilled water were first charged into the reactor, followed by emulsifier and stirred for 30 minutes. The polymerization reaction started just after initiator feeding, and the reaction continued for 4 hours. During polymerization, the stirring speed and polymerization temperature were maintained between 700 to 750 rpm and 70°C under constant nitrogen supply at 3 LPM. After the reaction reached completion, the mixture was filtered, and the resultant precipitation was dried at 60°C in a vacuum oven for 5 hours. Recipe for emulsion polymerization of acrylate colloid was given in Table 1.

Table 1. Recipe for emulsion polymerization of acrylate colloid

water	180 ml
MMA	106 ml
Sodium state	2 g
KPO	1 g
N ₂ gas	supply at rate 3 LPM
temperature	70°C
stirring speed	between 700 – 750 rpm

2.3. Fabrication of Colloidal Elastic Film

Natural rubber latex, PVL, was mixed acrylate particles in a ratio 1:1 under electric stirring to obtain composite latex. The mixture was stirred below 40 rpm until a homogenous blend was obtained. The freshly prepared acrylate-latex mixture was then placed into a rectangular glass substrate with a dimension of (210 x 148) mm for casting. The colloidal latex film was obtained by casting the mixture using a drawdown rod to obtain an acrylated latex film of uniform thickness. The cast was left to dry overnight under laboratory conditions, yielding a thin, transparent film. The thickness of the acrylated PVL film produced was \approx 500 μ m.

2.4. Characterization

Top surface images of colloidal acrylate latex film produced were acquired using scanning electron microscopy (SEM) with a field-emission scanning electron microscopic (Oxford Instrument) operated at

20kV. During this analysis, the film was cut in the dimension of 10 mm x 10 mm and placed in a sample holder for scanning to takes place. Since rubber is a non-conductive material, the film was coated first with a thin layer of gold in order to prevent charging or accumulation of static electric field at the specimen due to the electron irradiation required during imaging.

X-Ray Diffractometer (XRD) was used to analyse phase orientation stretched colloidal acrylate latex film. In this analysis, sample film was cut in the dimension of 20 x 10 mm and placed on the sample holder. The same film was then stretched to 200% elongation to obtain phase of oriented chain of the stretched film. Data obtained from both un-stretched and stretched analysis was compared and discussed.

2.5. Wavelength tunability of highly oriented colloidal acrylate latex film by changing lattice distance

Reflection spectra were obtained using an Ocean Optics mini spectrometer (USB 4000-VIS-NIR) and standard whiteboard as a reference. The 50x10mm stripe of sample was highly oriented by home-made device consisted of a tensile tester (INSTRON) to elongate the film, two different lenses for focusing the light beams and a mini spectrometer to measure the reflectance wavelength. The optical analysis was performed in the following manner. First, both ends of the film were positioned between the two grips of the tensile tester machine and stretch at rates of 500mm/min. Such a setup results in highly oriented acrylate spheres due to the high elongation of the stretched film (Figure 1). While stretching, an incidence light was directly introduced to the sample. Digital photographs of the film were taken using a digital SLR camera (CANON, EOS 7D). The reflectance wavelength, percent elongation during stretching were measured and recorded.

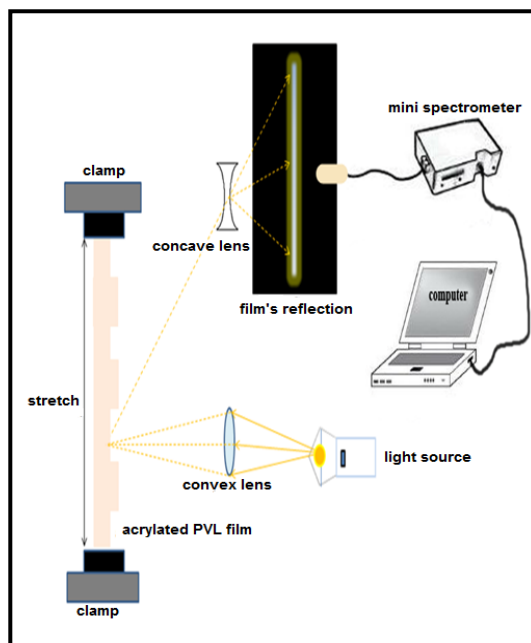


Figure 1. Wavelength tuning of highly oriented colloidal acrylate latex film by stretching.

3. Results and Discussion

3.1. Structure

This colloidal acrylate latex film consists of acrylate spheres arranged along the film. The spaces between one spheres to another is filled with PVL, thus forming a reversibly elastic film (Figure 2a and b). This make it is easier to reversibly deform the film by oriented the film into high elongation. High orientation by tensile tester during stretching, resulting in orderly arrangement of acrylate spheres along stretching direction. Acrylate spheres that are arranged on the surface of a latex film may create a grating spatial period, Λ , which can easily be described as the spacing distance between the spheres. The purpose of introducing grating into an elastic cavity is to integrate a wavelength-selective structure that is responsible for tuning the colour of photonic crystals under strain upon interaction with light (Yeh, 1994). Therefore, when the film is highly oriented, acrylate spheres may be arranged in an orderly manner according to the stretching direction and then become closer to one another (Figure 2c). When light is introduced to the stretched film, the reduction in the sphere distance may reflect light at a shorter

wavelength (Figure 2d). The wavelength values may change linearly with the sphere distance. As the spheres get closer, the wavelength gets shorter and vice versa (Figure 2e).

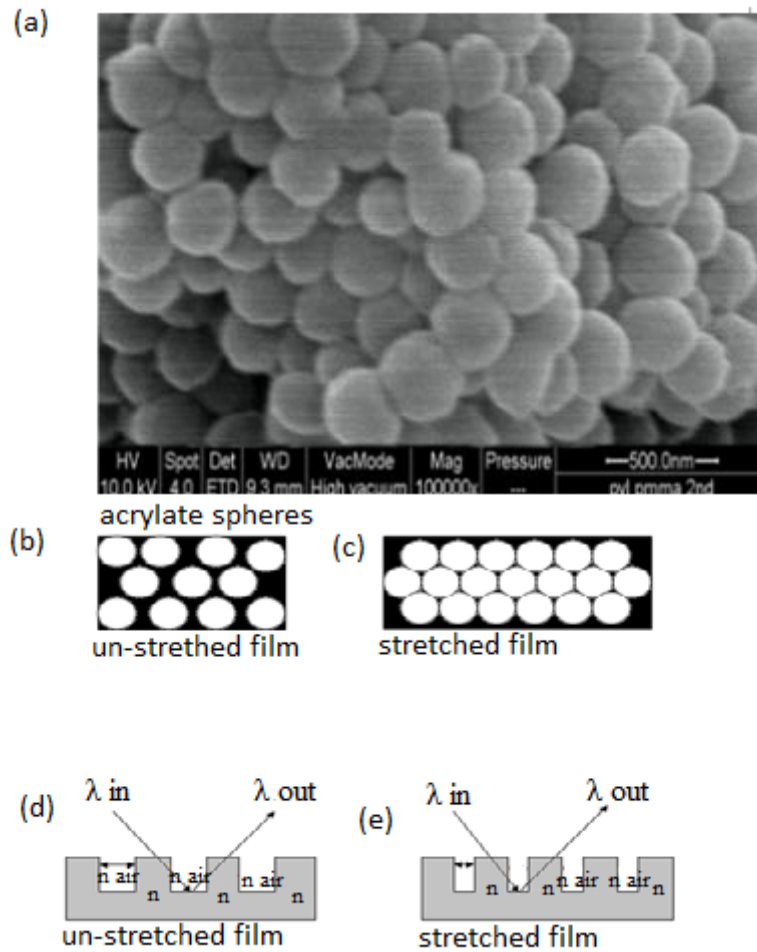


Figure 2. Colloidal acrylate latex film (a) SEM image (b) and (c) Transverse diagram of un-stretched and stretched film. (d) and (e) Grating's spatial period (Λ) that create wavelength-selective structures when the film is introduced to light.

The colloidal acrylate latex film was further analyzed using XRD to study the relationship between molecular orientation and spacing distance of the acrylate with wavelength responses. XRD was also used to determine the crystal plane by assigning the miller indices (hkl) for acrylate spheres. Analysis was performed under two different conditions such that a comparison of both patterns could be made. In the first case, analysis was performed using the un-stretched film, and the second case involved stretching of the same film to 200% elongation, with both ends being clamped into the position before scanning. The XRD patterns for both cases (un-stretched and stretched) are presented in Figure 3a and b. In the un-stretched case, there is only a single peak located at $29.376^\circ 2\theta$, which is identified as acrylate based on the standard XRD reference data base (ICDD, 2010, PDF 13-0835). No peaks are observed for rubber, which exhibits amorphous behavior in the un-stretched state (Toki et al., 2003). Therefore, it can be concluded that the sharp and narrow diffraction peaks indicate the molecular orientation of the rubber film during stretching. The relationship between strain and the orientation of the rubber chain can be further explained as the enhancement of the chain mobility in the amorphous region (Kameda and Asakura, 2003). Analysis of the peak leads to the conclusion that a highly oriented rubber chain begins to appear at some point between 13.899° , 16.379° , 17.938° , 20.626° and $25.009^\circ 2\theta$ (Baboo et al., 2010; Baboo, Sharma and Saxena, 2011) due to the elongation of the colloidal acrylate latex film. Peaks at these points can be attributed to the molecular orientation of the rubber chain that started to uncoil when the film was stretched.

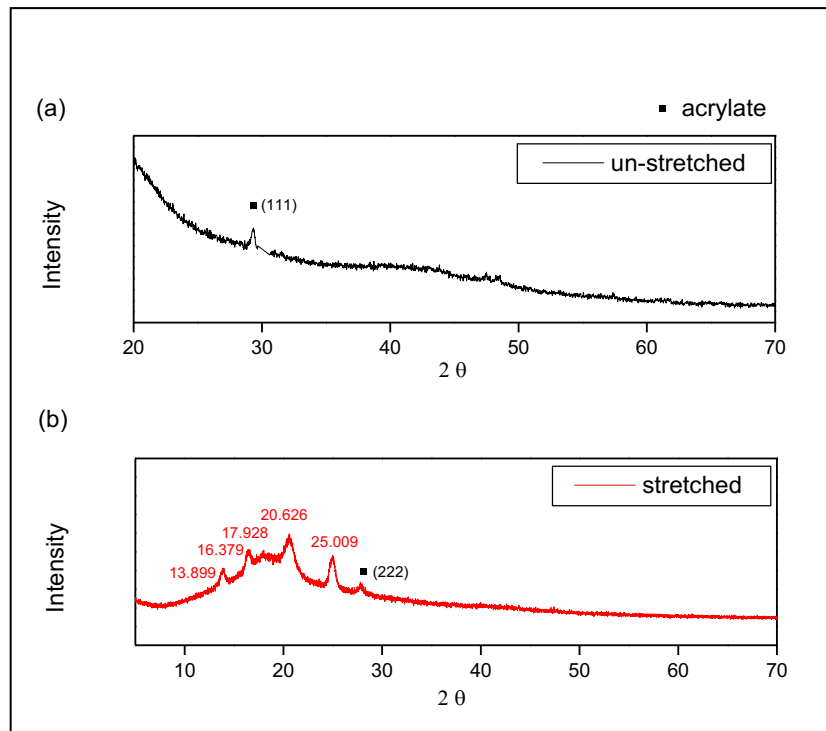


Figure 3. XRD pattern of (a) un-stretched colloidal acrylate latex film and (b) stretched colloidal acrylate latex film.

To prove that the reduction in the sphere distance affects the reflectance wavelength of the stretched film, the value of d at 200% elongation is calculated using Eq. (1).

$$d_{hkl} = D \sqrt{2/h^2 + k^2 + l^2}, \quad (1)$$

where D is the average diameter of the acrylate particle spheres.

In this study, the average diameter, D , of the acrylate spheres obtained from SEM analysis was approximately 513 nm. Meanwhile, the hkl miller indices for acrylate in the un-stretched and stretched state obtained from the XRD analysis were (111) and (222), respectively. Thus, the d spacing values of acrylate calculated from equation (1) are 419 nm for the un-stretched state and 209 nm when stretched to 200% elongation. From this calculation, it is proven that d decreases when the film is stretched to 200% elongation. Similar findings were reported by previous researchers, where the reflection stop band position decreased with an increase in the percentage of applied stretching due to decreases in the spacing distance parallel to the film surface (Fudouzi, 2009; Ying and Foulger, 2009). Therefore, it can be concluded that colour changes of the reflection are due to the decrease of the spacing distance of acrylate with respect to elongation of the film.

3.2. Wavelength tunable properties of highly oriented colloidal acrylate latex film

The reflection spectra at normal incidence of highly oriented colloidal acrylate stretched film were measured using a mini spectrophotometer and showed in Figure 4. While stretching, it is found that reflectance wavelength was tuned at three different strain levels which are 200%, 400% and 600%. The first reflectance wavelength at 200% elongation was 584.25nm. The second peak, 560.92nm was measured at 400% strain and the last peak of 467.47nm was measured at 600% strain. Thus, it can be said that mechanical deformation such stretching may have a profound effect on the reflectance wavelength. The relationship between the strain level (%) with the reflectance wavelength (nm) of highly oriented colloidal acrylate latex film is tabulated in Table 2.

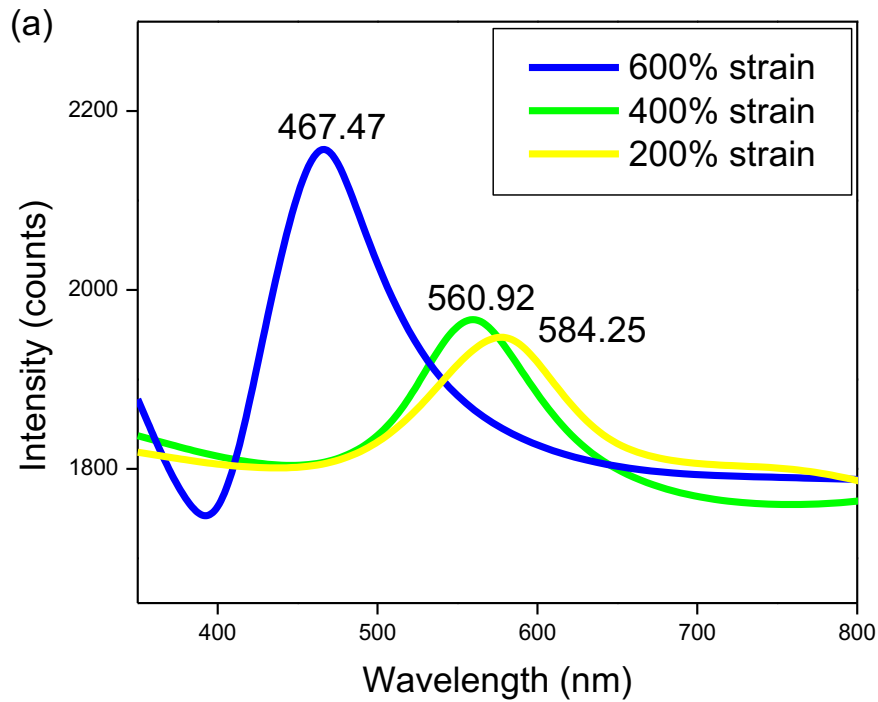


Figure 4. Reflection spectra of highly oriented colloidal acrylate latex film at different strain level.

Table 2 Relationship between the strain levels with reflectance wavelength of highly oriented colloidal acrylate latex film.

Strain Level (%)	Reflectance Wavelength (λ)
200	584.25
400	560.92
600	467.47

In order to evaluate the wavelength tunabilities properties of colloidal acrylate latex film fabricated, their reflectance wavelength at various strain levels recorded were compared with the literatures. It is found that none of the strain levels measured in this analysis is similar to the findings reported by previous researchers. In fact, the closest strain values that gives almost similar findings is only range of wavelength between 450 to 495 nm which recorded at 600% strain in this research and 530% strain by Sun et al. (2012). In addition, tensile strain required to tune the reflectance wavelength of colloidal acrylate latex film produced in this research is higher than those obtained in the literatures. For example, Wohlleben et al. (2007) demonstrated reflectance wavelength of yellow colour (570 – 590 nm) at only 14% strain, green (495 – 570 nm) at 28% strain and blue (450 – 495 nm) at 60% strain. Similar results were reported by Fudouzi and Sawada (2006) where the film fabricated exhibited reflectance wavelength of 570 nm at 15% strain and shifted to 560 nm at 34% strain. Table 3 summarizes comparison between results obtained in this study with other literatures. Comparison is done in term of material used and strain needed for wavelength response to occur. Close observation on the results leads to a conclusion that periodicity and monodispersity of spheres used are the most important aspects in determining reflectance wavelength of the stretched film. A periodic arrangement of high refractive index contrast in photonic crystal structure allows better observation of the resultant reflectance colour (Kolle, 2011). In this research, refractive index contrast between PVL and acrylate spheres is relatively low, therefore high elongation is needed to create alternating arrangement of high and low refractive indices along the film. As an alternative, an inverse opal structure can be fabricated to produce colloidal crystal structure with high refractive index contrast. It can be fabricated by removing spheres to create air voids in the structure. The voids then will be filled with other material of high refractive index to produce a photonic structure with large refractive index contrast. A research done by Sumioka, Kayashima, and Tsutsui (2002) reveals that inverse opal structure gives a shift to a shorter wavelength in comparison to opal structure due to the existing of high effective refractive index of the medium.

Table 3. Relationship between the strain levels with reflectance wavelength of highly oriented colloidal acrylate latex film

Material	^a Colloidal monodisperse acrylate spheres & PVL		
Colour	Blue	Green	Yellow
Wavelength (nm)	467	560	584
Strain (%)	600	400	200
Material	^b Polymethylmethacrylate (PMMA) spheres & Butyl acrylate		
Colour	Blue	Green	Yellow
Wavelength (nm)	475	570	590
Strain (%)	530	120	117
Material	^c Monodisperse core shell polymer beads		
Colour	Blue	Green	Yellow
Wavelength (nm)	450 – 495	495 – 570	570 – 590
Strain(%)	60	28	14
Material	^d Colloidal monodisperse polystyrene & polydimethylsiloxane		
Colour	Blue	Green	Yellow
Wavelength (nm)	Not reported	560	570
Strain (%)	Not reported	34	15

^aResults obtained in this study

^bC. Sun et al²²

^cWohlleben et al⁴⁰

^dFudouzi and Sawada²⁶

Under an applied stress by tensile tester machine, colloidal acrylate latex film is reversibly undergone stretch and release step for two cycles. Elastic characteristic of PVL make it is possible to stretch and un-stretched the film repeatedly. By using a mini spectrometer, wavelength changes due to applied strain were recorded. Figure 5 (a) and (b) depicts the spectra results of highly oriented colloidal acrylate latex film under 200, 400 and 600 % strain levels corresponding to stretching in vertical direction. At first cycle, the reflectance wavelength was observed to recover almost of all initial positions with a slight reduction in wavelength values during the stretching and releasing cycles. At second cycle, the film was highly oriented again from 200% until 400% strain. As expectedly, the film shows reduction in reflectance wavelength with the increase of strain level. The film was then recovering its reflectance wavelength when the strain was reduced back to 400% and 200% strain. It is found that, for every cycle, the new reflectance wavelength for each strain level is slightly lower than the previous one. These changes provided evidence that the wavelength has ability to return to its original position but not in the exact position as it were before. From stretch and release step, it is found that colour reflection of colloidal acrylate latex film can be reversibly changed by stretching. Moreover, the elasticity of the rubber will give immediate recovery of the film when the strain force was released. This finding indicates that the wavelength of highly oriented colloidal acrylate latex film can be reversibly tuned using different elongations upon stretching and releasing.

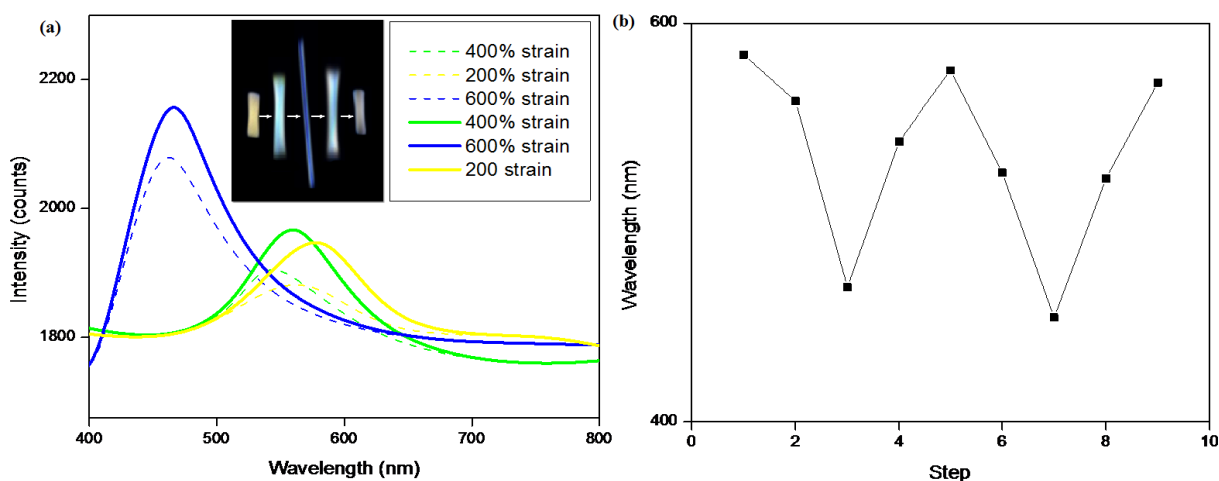


Fig. 5 Wavelength tunability of highly oriented colloidal acrylate latex film (a) Reflection spectra of film at 200, 400 and 600% strain level. (b) Stretch-release step of the film for 2 cycles.

3.3. Bragg-Snell Law

From the results obtained during wavelength tunability analysis, it can be clearly seen that when a tensile stress was applied to the colloidal acrylate latex film, the reflection wavelength shifted to a shorter wavelength. This agrees well with the Bragg- Snell Law as shown in Eq. (2).

$$\lambda = 2d \sqrt{n_{eff}^2 - \sin^2\theta}, \quad (2)$$

where λ is the reflectance wavelength, d is the spacing distance of the spheres, n_{eff} is the effective refractive index, and θ is the angle of incidence light on the sample along the [111] direction. In a closed packed structure, the effective refractive index, n_{eff} , is given by $n_{eff} = n_{polymer} f_{polymer} + n_{air} (1 - f_{polymer})$ (Ge et al., 2006). Theoretically, upon stretching, the spacing distance of the spheres (d) is decreased and light will be diffracted to a shorter wavelength. Therefore, it can be said that wavelength values change linearly with the value of spacing distance. In this analysis, when colloidal acrylate latex film is highly oriented into some extent, d will decrease due to the film elongation. Hence, when an incidence light is introduced on the stretched film, the light will be reflectance to a shorter wavelength with increasing strain level. In contrast, when the strain force of the same film is released, the wavelength will automatically return to its original position. This process is a reversibly process as we can simply tune the reflectance wavelength of the film by manipulating the sphere distance by strain deformation.

4. Conclusion

In conclusion, we presented that self-assembly of colloidal acrylate spheres and pre-vulcanized latex can be used in the fabrication of wavelength tunability material. By highly oriented the colloidal acrylate latex film at different strain levels, the reflectance wavelength shows a reduction from 584.25 to 560.92 and 467.47 nm. Result obtained by this research, can be used as a basis in the development of rubber latex in strain sensing application. We note that further improvement during fabrication and optical analysis need to be improved for better results.

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