



# Preparation of Panchromatic Silver Halide Emulsion to Fabricate High Efficiency Multiplexed Volume Phase Holograms for Solar Concentration Application

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**Abstract:** Volume Phase Gratings (VPG) are often recorded using holographic emulsions such as photopolymer, Dichromated Gelatin (DCG), and silver halide. We employed silver halide emulsion to record the VPG in monochromatic red wavelength for solar concentration purposes. According to the Braggs condition, the recorded VPG's diffraction efficiency reaches its peak at the recorded angle and wavelength. For solar applications, we needed high diffraction efficiency over a wide angle and wavelength range. So, we captured multiplexed single VPG in our own panchromatic silver halide emulsion. The procedure of making panchromatic silver halide emulsion is described in depth. This technique is unique in that it achieves peak diffraction efficiency at broad wavelength and wide angle in a single multiplexed VPG. The panchromatic multiplexed VPG produced a high diffraction efficiency in wide-angle response with overall visible wavelengths. This study describes the high diffraction efficiency multiplexed VPG in its own manufactured panchromatic silver halide emulsion for solar concentrating applications.

**Keywords:** Silver Halide Emulsion, Holographic Optical Elements, Volume Phase Grating, Diffraction Efficiency, Photovoltaic Cell, Solar Concentrator

## 1. Introduction

Nowadays, Photo Voltaic (PV) cells play a vital role for generating electricity. However, most of the PV cells are having limited output efficiency [1–7]. To enhance its output efficiency, several PV cells are connected in a solar panel system but the main drawback is that the PV cells are very expensive. In order to avoid expensive PV cells, the conventional solar concentrating system was introduced by using of reflecting mirror and focusing lens with tracking mechanism [8-11]. But the IR radiation in the form of heat is highly affecting the PV cells self-life. Then the technology of Holographic Solar Concentrating (HSC) system was introduced with cool light concentration which improves PV cells life with self-tracking mechanism [12-20]. The HSC idea has been introduced since the early of 1980's but still it has limited success. Although, the company named Stone Ridge applied HSC successfully. Photopolymer and DCG emulsions are the main holographic materials used to record Holographic Optical Elements (HOE) for various type of outdoor applications [21-30]. In this work we have used

silver halide emulsion for HSC application. The silver halide is the first ever material used for holography with numerous applications. The silver halide material has great advantages of high diffraction efficiency, high transparency, high resolving power, long term stability, low noise ratio, and high sensitivity [31-33]. But the main drawback of this material is high cost and limited availability. Hence, we have prepared our own silver halide emulsion (monochromatic and panchromatic) followed by these reference works [34-36]. The emulsion layer thickness and grain size are the two critical parameters, which defines the diffraction efficiency and output noise ratio of the recorded hologram. There are different types of HOE's used for solar concentrating applications such as holographic gratings, holographic reflection elements, holographic transmission elements and multiplexed HOE's etc [37-39]. In present work we fabricate the monochromatic VPG and studied its angular and wavelength responses. Due to Braggs condition the holograms can't deliver high diffraction efficiency on broad wavelength and wide angle. So, an external device needs to track the suns movement for large angle coverage. To overcome this issue

multiplexed method has been introduced as self-tracking device. But, lower diffraction efficiency, complex recording method and large interlayer crosstalk are some drawbacks of the multiplexed method [40, 41]. Here, we have proposed a novel design of multiplexed VPG to overcome all those issues.

## 2. Experimental Section

### 2.1. Experimental arrangement for monochromatic red sensitive VPG

Basically, there are two types of recording methods (reflection and transmission) were used in holography. In this work we have made transmission type arrangement for the VPG recording. The monochromatic red sensitive silver halide emulsion is the recording material. The He-Ne 632.8 nm emitting laser is the source which is used for VPG recording. The Electronic Shutter (ES) was used to control the recording time of the hologram. A single laser beam divided into two parts with the help of density variable Beam Splitters (BS). Object beam redirected from the front coated aluminum Mirror (M1) to Spatial Filter (SF1), and Collimating Lens (CL1) then monochromatic red sensitive Holographic Plate (HP). The reference beam from BS to Mirror (M2) and Spatial Filter (SF2) and Collimating Lens (CL2) then monochromatic red sensitive HP. Angle between the reference beam and object beam is  $21^\circ$  is arranged. The schematic diagram of the recording geometry of red sensitive VPG is shown in Figure.1. (a). Both laser beams are interfered at front surface of HP to form transmission type VPG. And the top view of experimental arrangement is shown in Figure.1. (b), the recorded red sensitive VPG is shown in Figure.1. (c). Total experiment setup placed on top of vibration isolation table. The experiment was carried out as trial-and-error method to achieve high diffraction efficiency holograms.

The sensitivity of monochromatic silver halide emulsion was inspected by exposed energy. We got

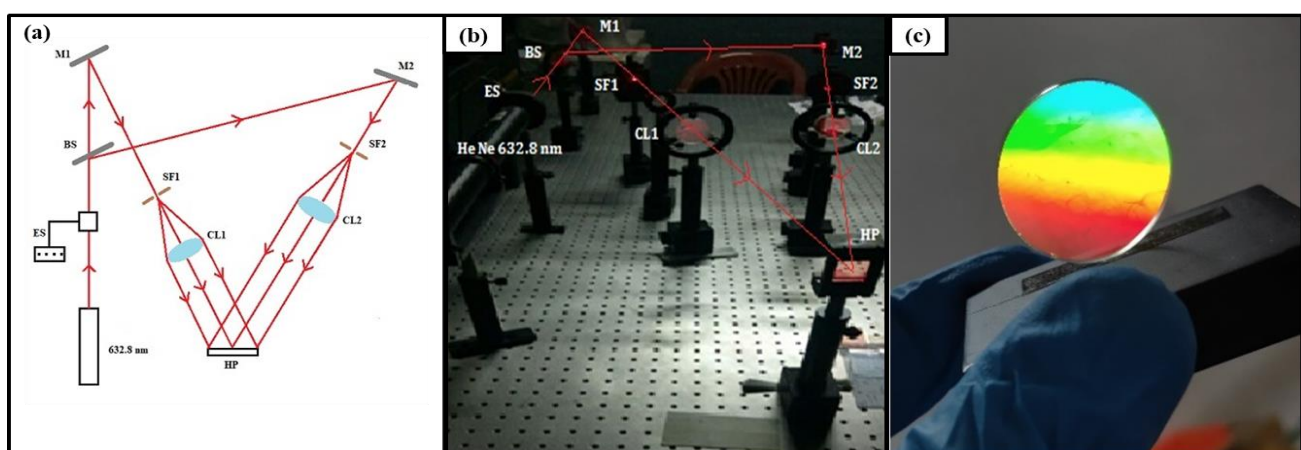
peak diffraction efficiency of 65% and calculated exposure time is 1 minute 45 seconds. The commercial chemical developer (CW-C2) was used for the wet processing technique. Experimental observations revealed that the suitable developing time is 2 minute 20 second and bleaching (PBU-type) time is 2 minutes with mild agitation. The running De-Mineralized (DM) water was used for washing plates which is about 3 minutes and left for natural drying. The recorded hologram is cemented immediately to protect from physical damage and moisture.

### 2.2. Panchromatic silver halide emulsion preparation

Quality float glass plates are preferred as the substrates for silver halide emulsion coating. Properly un-cleaned glass substrates are tending to peel off while after coating or developing. Therefore, highly cleaned glass substrates are used for the emulsion coating. To obtain better result, the glass substrate put in to the Hydro Chloric Acid (HCL 80%) bath for 12 hours. And for final washing we used house hold dishwasher and De-ionized (DI) hot water which gives the better result.

#### 2.2.1 Emulsification

To prepare emulsion more attention is given on gelatin concentration, layer thickness and spectral sensitivity. Selection of gelatin type (photographic grade) is another important factor to obtain good results. There are three solutions prepared and named as solution A, B and C. Solution A contains 2% of gelatin - water combinations and solution B and solution C contains silver - halide components. Solution B and solution C were mixed with solution A to form silver halide emulsion by using magnetic stirring. The silver halide emulsion general formula is given in the eq.1.



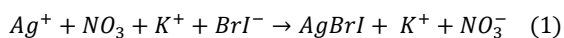
**Figure1.** (a) Recording geometry of monochromatic red sensitive VPG (b) Top view of experimental arrangement (c) Recorded monochromatic red sensitive VPG

Were,

Solution A – 2gm gelatin + 100 ml DI water

Solution B – 2gm silver nitrate + 40 ml DI water

Solution C – 1.5gm potassium bromide + 0.5gm potassium iodide + 40ml DI water



### 2.2.2 Repining

In order to control the grain size, we used double-jet mixing method. Solution B (silver) and solution C (halide) added in to solution A (gelatin). During mixing the clear liquids turns into milky white to form silver bromo-iodide solution called as precipitation. At this stage the sensitivity of the emulsion slightly increased, and it is important to keep the solution temperature 38°C for 20 minutes under continuous stirring. Temperature of the solution (38°C) and mixing time (50-80 sec) are the crucial parameters; these are maintained for the best results. Nucleation method was carefully handled during emulsification to achieve best silver halide emulsion. Hence, the filtered emulsion shifts immediately into refrigerator under 7°C (nucleated state) to prevent from grain growth.

### 2.2.3 Washing

After 12 hours the stiff jelly emulsion takes out from the refrigerator and makes it into noodles. The cold DI (15°C) water was used for washing the emulsion to remove the excess salts and other impurities.

### 2.2.4 Sensitizing

Properly washed emulsion re-heated up to 40°C under hotplate magnetic stirrer then added of red and green sensitive dyes (cyanine) to improve the spectral sensitivity up to panchromatic range. At this stage wetting agent, hardener and anti-bacterial preservatives were added.

### 2.2.5 Coating

Airflow coating table made up with zero angle tilt alignment, Relative Humidity (RH) 40% and 15°C room temperature was maintained. Coating substrates are paste with transparent tape on four corners to prevent the emulsion overflow under glass substrates. Before coating the glass substrate surface slightly heated to avoid peel off issues. To maintain coating thickness and surface evenness the Mayer Bar method with optimized winding were used (28, 30, 32, 34 wire gauge). The 32-wire gauge is the best selection to maintain emulsion coating thickness of 10-microns for the panchromatic multiplexed VPG purpose. The re-heated emulsion pours on top of the coating glass substrate and slowly draw down by Mayer Bar (32 wire gauge) with constant force. The emulsion coated plates were left for 24 hours drying at 15°C temperatures.

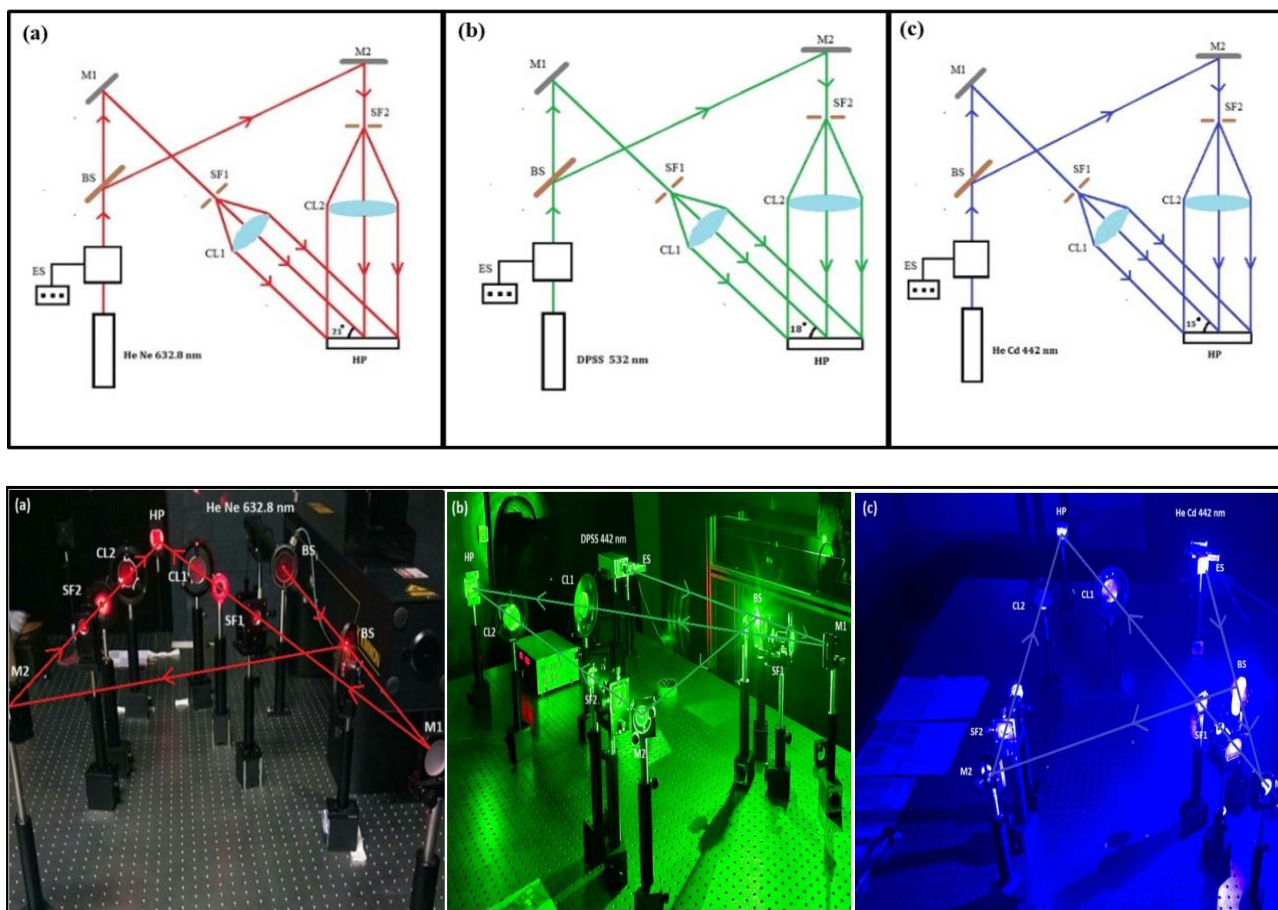
## 2.3. Experimental arrangements for panchromatic multiplexed VPG

The panchromatic silver halide emulsion is used to record multiplexed VPG in three different wavelengths (He Ne 632.8 nm, DPSS 532 nm and He Cd 442 nm) on three optical table arrangements. The experimental arrangement for the same is shown in Figure. 2. (a) The first setup arrangement is made up with He Ne 632.8 nm laser source and optimized recording incident angle is 21°. The second setup arrangement is Figure. 2. (b) made up with DPSS 532 nm laser source with incident angle is 18°. And the third setup arrangement is Figure. 2. (c) made up with He Cd 442 nm laser source and the incident angle is 15°.

Totally three recordings were done with three selected angles with selective wavelengths in a single panchromatic silver halide emulsion, it is a challenging part in this experiment. These three selective angles and wavelengths (632.8 nm -21°, 532 nm -18° and 442 nm -15°) are obtained from monochromatic VPG diffraction efficiency results. The multiplexed VPG's recording angle, wavelength and exposing time is mentioned in Table.1.

**Table 1.** Multiplexed VPG recording angle, wavelength, exposure time and exposure energy

Multiplexed VPG Recording	First shoot	Second shoot	Third shoot
Incident angle	21°	18°	15°
Wavelength	632.8 nm	532 nm	442 nm
Exposure time	65 sec	80 sec	115 sec
Exposure energy	160 μJ/cm <sup>2</sup>	230 μJ/cm <sup>2</sup>	1100 μJ/cm <sup>2</sup>



**Figure 2.** (a) Recording geometry and recording arrangement of panchromatic multiplexed VPG by (a) 632.8 nm (b) 532 nm (c) 442 nm

**2.4. Diffraction efficiency measurement setup**

The wavelength response and angular response of the recorded panchromatic multiplexed VPG measured by three various laser sources by placing on angle rotating stage is shown in Figure. 3. (a) 632.8 nm (b) 532 nm (c) 442 nm. The diffraction efficiency is ratio between the power of diffracted beam and incident beam of the hologram is given in the eq.2.

$$D\eta = DI/D0 \tag{2}$$

Diffraction efficiency = Input diffracted beam power / Output diffracted beam power

The first setup is made up of He Ne 632.8 nm laser source with angle rotating stage. The input power can be measured of the direct laser beam without VPG. For output power measurement, the panchromatic multiplexed VPG is placed on top of the angle rotator and allow the laser beam to hit on the VPGs front surface. The left and right side first order diffracted beams are projected behind the hologram. The diffracted beams are capture by the photometer (Vega Ophir power meter). The output diffraction efficiency of the VPG has been taken from the angle 10<sup>0</sup> to 30<sup>0</sup>. Same procedure followed for the 532nm and 442nm wavelength emitting laser source measuring setups.

**3. Results and discussion**

**3.1. Diffraction efficiency and angular responses of monochromatic red sensitive VPG in three different wavelengths**

The diffraction efficiency measurement of monochromatic red sensitive VPG is carried out. The angular and wavelength response of monochromatic red sensitive VPG in three various wavelengths (He Ne 632.8 nm, DPSS 532 nm and He Cd 442 nm) are studied. According to the Braggs condition the obtained peak diffraction efficiency of monochromatic VPG is 66% at 21<sup>0</sup> in red wavelength (632.8 nm), 51% at 18<sup>0</sup> in green wavelength (532 nm) and 41% at 15<sup>0</sup> in blue wavelength (442 nm) [42]. The diffraction efficiency comparison of monochromatic VPG is shown in following tables and figures. Table 2. is the monochromatic VPG's diffraction efficiency with angular response for three selective wavelengths left side first order measurement readings. Table. 3. is the monochromatic VPG's diffraction efficiency with angular response for three selective wavelengths right side first order measurement readings. The Figure. 4. Represent the VPG's left side first order values and Figure. 5. Represent the VPG's right side first order values.

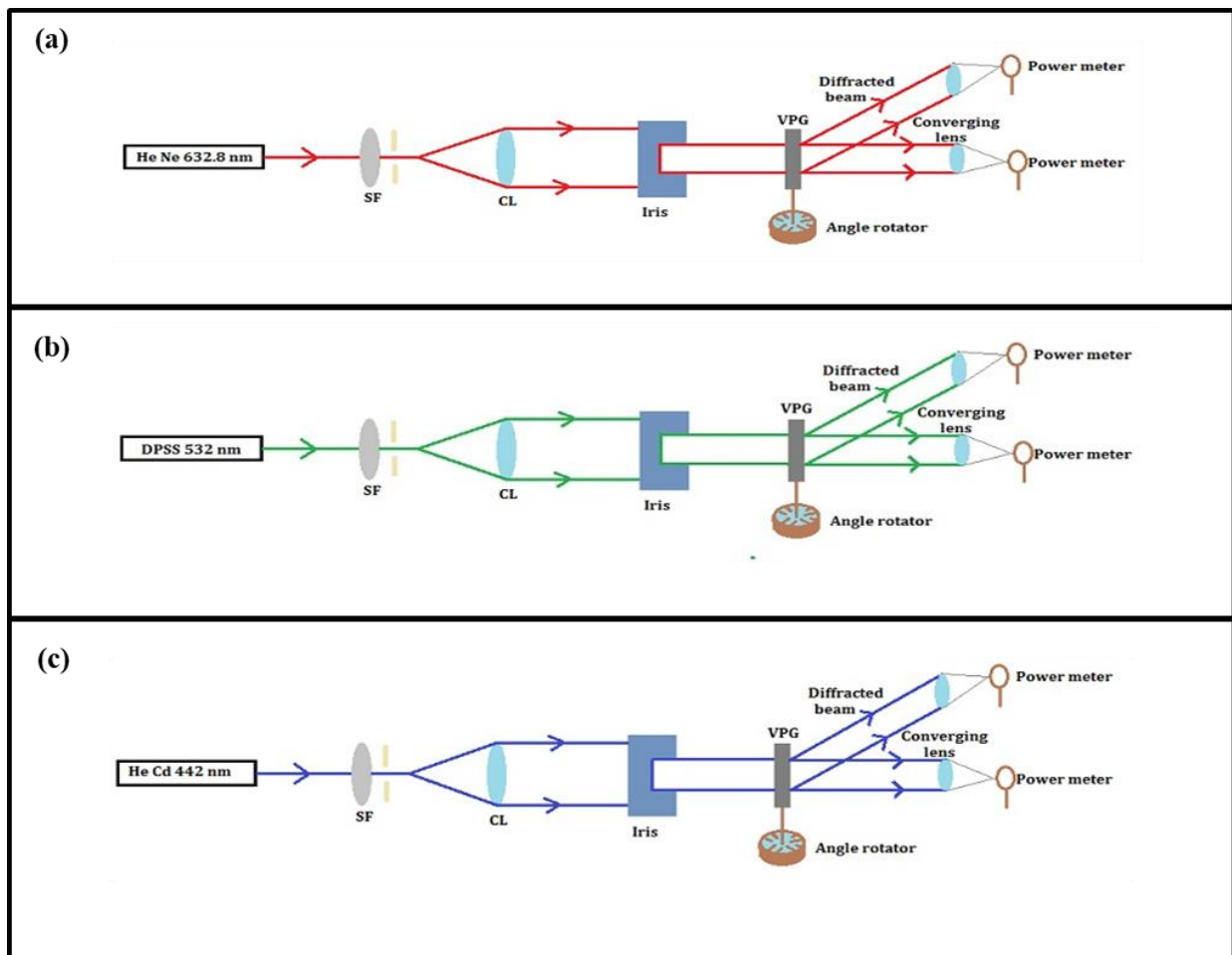


Figure 3. VPG diffraction efficiency measuring setup with three various laser sources (a) 632.8 nm (b) 532 nm and (c) 442 nm

Table 2. Monochromatic red sensitive VPG's diffraction efficiency and angular response in three selective wavelengths left side first order measurement readings

Incident angle	Diffraction efficiency 632.8 nm	Diffraction efficiency 532 nm	Diffraction efficiency 442 nm
10°	17%	19%	19%
12°	29%	25%	29%
14°	35%	31%	33%
15°	41%	37%	<b>40%</b>
16°	45%	43%	34%
18°	51%	<b>51%</b>	29%
20°	57%	46%	24%
21°	<b>65%</b>	39%	18%
22°	60%	33%	14%
24°	53%	26%	9%
26°	45%	21%	5%
28°	33%	15%	2%
30°	21%	9%	1%

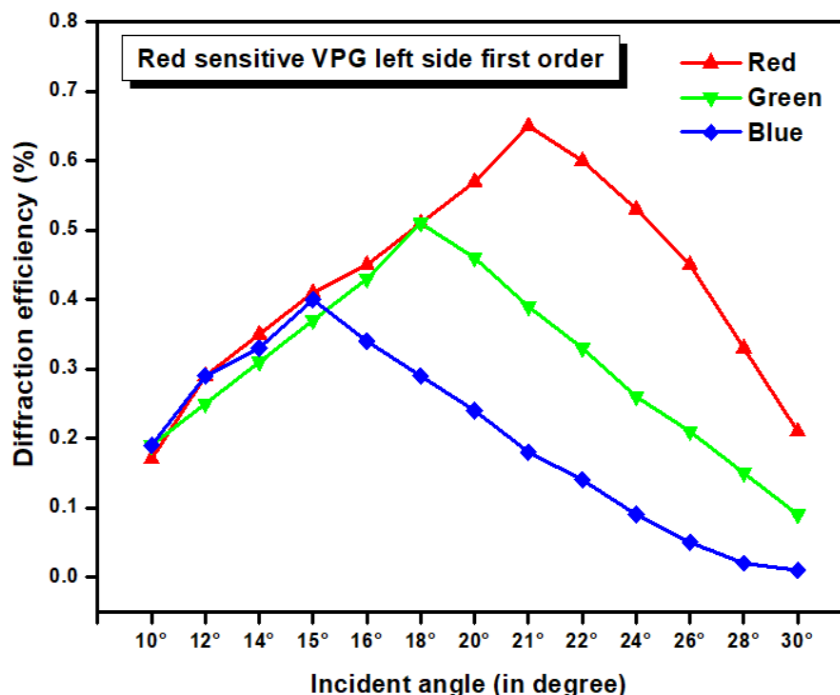


Figure 4. Monochromatic red sensitive VPG’s diffraction efficiency and angular response in three selective wavelengths of left side first order values

Table 3. Monochromatic red sensitive VPG’s diffraction efficiency and angular response in three selective wavelengths of right side first order measurement readings

Incident angle	Diffraction efficiency 632.8 nm	Diffraction efficiency 532 nm	Diffraction efficiency 442 nm
10°	21%	20%	18%
12°	36%	29%	29%
14°	40%	34%	34%
15°	45%	41%	41%
16°	50%	45%	35%
18°	54%	50%	31%
20°	61%	46%	27%
21°	66%	37%	20%
22°	60%	30%	17%
24°	55%	27%	11%
26°	47%	21%	7%
28°	36%	18%	4%
30°	27%	10%	2%

### 3.2 Study on diffraction efficiency against thickness of the prepared panchromatic emulsion

The important parameter of coated emulsion thickness is playing a big role in diffraction efficiency of the final hologram [43, 44]. For monochromatic

recording the emulsion thickness is 6-8 microns enough to achieve best holograms. But in multiplexing method more thickness of the emulsion layer is necessary to achieve high diffraction efficiency holograms. Hence, we coat the emulsion thickness from 6-14microns by optimized winding Mayer Bar (28, 30, 32, 34 wire

gauge). We obtained higher diffraction efficiency of 76% against the thickness is 10microns by 32 wire gauge Mayer Bar is shown in Figure. 6.

### 3.3. Study on exposure energy and chemical treatment of panchromatic multiplexed VPG

The exposure energy and chemical developing times were analysed in order to achieve high diffraction efficiency panchromatic multiplexed VPG [45–48]. The

optimized exposing energy of the panchromatic emulsion is  $160\mu\text{J}/\text{cm}^2$  for red (632.8 nm),  $230\mu\text{J}/\text{cm}^2$  for green (532 nm) and  $1100\mu\text{J}/\text{cm}^2$  for blue (442 nm). The optimized chemical processing time is in-between 1 - 2.40 minutes. The high diffraction efficiency of 76 %is obtained at 2 minutes 20 seconds chemical processing. The experimental details of the given exposure energy against obtained diffraction efficiency is shown in Figure. 7. and its chemical processing time against achieved diffraction efficiency is shown in Figure.8.

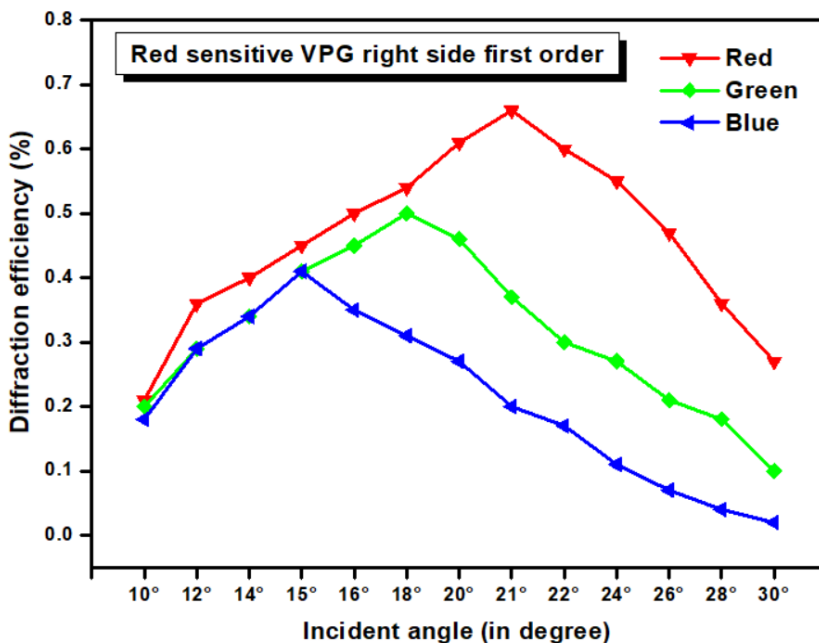


Figure 5. Monochromatic red sensitive VPG's diffraction efficiency and angular response in three selective wavelengths of right side first order values

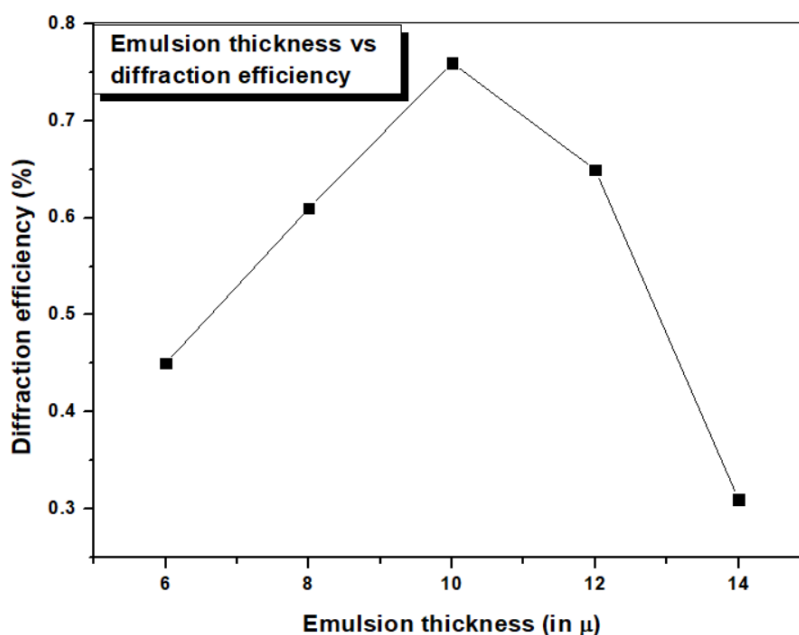


Figure 6. Panchromatic silver halide emulsion thickness against diffraction efficiency

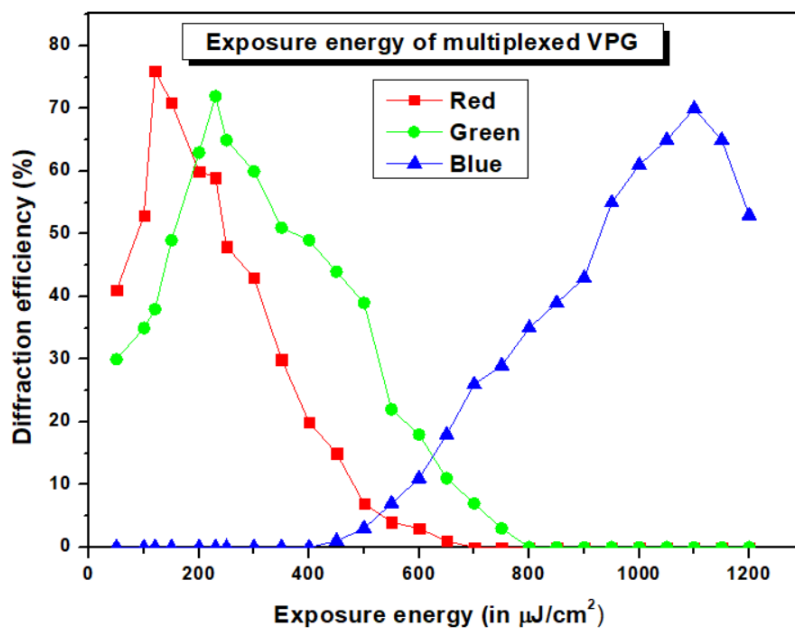


Figure 7. Exposure energy vs diffraction efficiency of multiplexed VPG

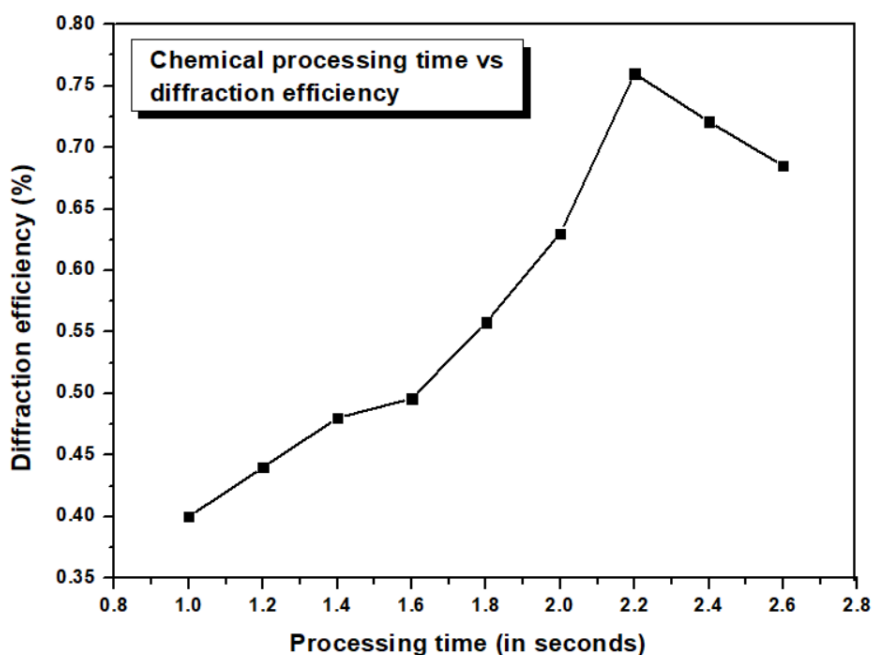


Figure 8. Chemical processing time vs diffraction efficiency of panchromatic multiplexed VPG

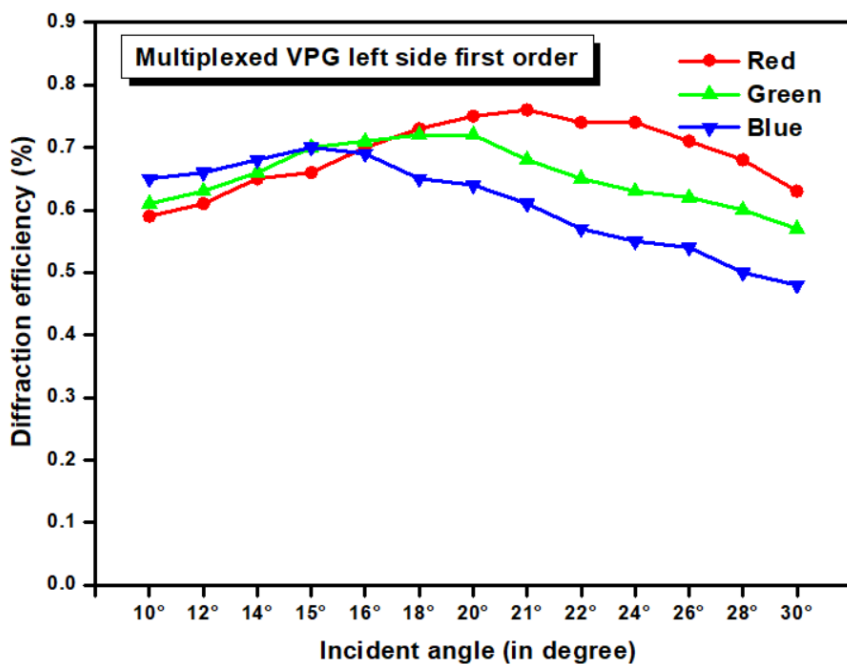
### 3.4. Diffraction efficiency and angular responses of multiplexed VPG in three different wavelengths

The diffraction efficiency of multiplexed VPG's angular and wavelength responses in three different wavelengths (He Ne 632.8 nm, DPSS 532 nm and He Cd 442 nm) are studied. The obtained high diffraction efficiency is 76% in 632.8 nm, 72% in 532 nm and 70% in 442 nm, left and right side first order readings are shown in Table 4 and Table 5. Respectively. The obtained diffraction efficiency proves a flat angle

response and overall visible wavelength coverage. The left and right side first order values are shown in Fig. 9. and Figure. 10. Respectively. Many of researchers worked to obtain high diffraction efficiency holograms in multiplexed method [40, 41, 43, 46]. But they didn't overcome with limited diffraction efficiency issue and large interlayer crosstalk issue. Here, we have fabricated the high diffraction efficiency multiplexed VPG with wide angular and broad wavelength responses by novel recording design, which will have a high impact on the solar concentrating applications to enhanced output efficiency of PV cells without heat energy.

**Table 4.** Multiplexed VPG’s diffraction efficiency and angular response in three wavelengths left side first order measurement readings

Incident angle	Diffraction efficiency 632.8 nm	Diffraction efficiency 532 nm	Diffraction efficiency 442 nm
10°	59%	61%	65%
12°	61%	63%	66%
14°	65%	66%	68%
15°	66%	70%	<b>70%</b>
16°	70%	71%	69%
18°	73%	<b>72%</b>	65%
20°	75%	72%	64%
21°	<b>76%</b>	68%	61%
22°	74%	65%	57%
24°	74%	63%	55%
26°	71%	62%	54%
28°	68%	60%	50%
30°	63%	57%	48%



**Figure 9.** Multiplexed VPG’s diffraction efficiency and angular response in three wavelenaths left side first order values

**Table 5.** Multiplexed VPG’s diffraction efficiency and angular response in three wavelengths right side first order measurement readings

Incident angle	Diffraction efficiency 632.8 nm	Diffraction efficiency 532 nm	Diffraction efficiency 442 nm
10°	56%	61%	63%
12°	60%	65%	66%

14°	65%	67%	67%
15°	66%	68%	69%
16°	68%	71%	68%
18°	72%	72%	65%
20°	75%	71%	61%
21°	76%	68%	58%
22°	74%	65%	55%
24°	71%	64%	53%
26°	67%	60%	52%
28°	65%	58%	49%
30°	63%	55%	47%

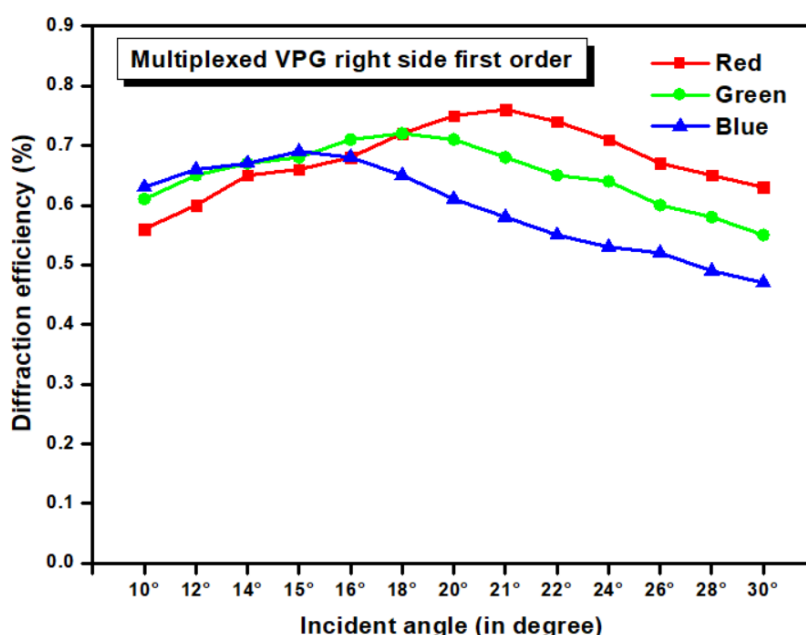


Figure 10. Multiplexed VPG's diffraction efficiency and angular response in three wavelengths right side first order values

### 5. Conclusion

In this present study, a monochromatic Volume Phase Grating by 632.8 nm laser source was fabricated for the solar concentrating application. According to the Bragg's condition the obtained peak diffraction efficiency of monochromatic VPG is only on recorded angle and recorded wavelength. In order to improve its diffraction efficiency in wide angular and broad wavelength, we approached a novel design multiplexed VPG in our own panchromatic silver halide emulsion. The multiplexing method has main drawback of lower diffraction efficiency, large interlayer crosstalk and complex recording methods. To overcome those issue, we prepared our own panchromatic emulsion and make it suitable for multiplexing VPG. The highly crucial parameters of emulsion preparation method, coating technique, emulsion layer thickness, recording

technique and chemical processing methods are optimized and workout a novel recording design. The obtained results of panchromatic multiplexed VPG has high diffraction efficiency in broad wavelength responding of 76% in 632.8 nm, 72% in 532 nm and 70% in 442 nm with wide angular response of 10°-30° in 10-micron thickness of emulsion layer. By the novel recording design of three wavelength and angle combination multiplexed recording we have overcome the interlayer crosstalk issue and complex recording issue. The obtained results proved a flat response of peak diffraction efficiency with broad angular and overall visible wavelength coverage multiplexed VPG. The developed novel design of multiplexed VPG can advantageously be used to enhance the output efficiency of Photovoltaic (PV) cells without eternal tracking device.

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### **Data Availability**

The data supporting the findings of this study can be obtained from the corresponding author upon reasonable request.

### **Has this article screened for similarity?**

Yes

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