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SILVER MICRO- AND NANO-PARTICLES OBTAINED USING DIFFERENT GLYCOLS AS REDUCING AGENTS AND MEASUREMENT OF THEIR CONDUCTIVITY

Article Highlights

- Silver micro- and nanoparticles were obtained by chemical reduction using glycols in the presence and absence of PVP
- Silver particles were used for the preparation of conductive pastes for the metallization of solar cells
- Powder properties were determined by XRD, SEM and DSC/TGA
- The obtained samples showed good crystallinity, purity and spherical morphology
- The conductive behavior of pastes is dependent on the size, shape and type related to the synthesis process

Abstract

Synthesis of silver micro- and nano-particles for the preparation of conductive pastes for the metallization of solar cells was realized by chemical reduction in the presence and absence of poly(vinyl-pyrrolidone) (PVP). Silver nitrate was used as a precursor in the presence of three polyols (ethylene glycol, di-ethylene glycol and propylene glycol) tested at experimental temperatures near their boiling points. Six samples were obtained by this protocol. Three silver powders obtained without the use of PVP has a metallic luster appearance; however, the samples produced using an excess of PVP were in the form of stable colloidal dispersions of silver nano-particles. Structural characterizations of samples using a scanning electron microscope and X-ray diffractometer showed good crystallinity and spherical morphology. From DSC and TGA analyses, it was observed that all the nano-silvers present in the colloidal suspension have the same thermal behavior.

Keywords: silver nitrate, nano-particles, chemical reduction, glycol derivatives, PVP.

The operating characteristics of photovoltaic solar cells depend upon their realization process and, more particularly, on the nature and quality of the front and rear electrical contact [1,2]. These contacts in the form of a grid are generally realized using screen printing technology. They consist of a homogeneous melted mixture of metallic oxides with additional chemicals [3]. Silver has the highest electrical and thermal conductivity among metals, making it a popular material for electrical contacts. Generally, silver powder is used as screen printed for making elec-

trical contacts on silicon solar cells due to its excellent properties [4]. The thick film conductive paste contains spherical and/or flake-form silver powder of very high conductivity, chemical stability, and low price.

In order to improve the layer adapted to the desired properties, good knowledge of the physical and morphological properties of silver powder is necessary. Given the range of applications in which silver is important, better control of its properties would have profound economic implications. It is well established that, by shrinking the size of a solid particle to the nanometer regime, the chemical, electrical, mechanical, and optical properties can be altered [2]. Silver, which represents the most important component in the paste, occurs in particles with various shapes and sizes. It must have suitable properties (surface

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area, particle size and distribution), which depend on the process parameters. Since silver is the main component influencing the metallic contact, the smaller are its particles sizes the greater is its specific area in the powders with the best characteristics; the specific area being the main factor favoring the film continuity [4].

To manufacture these powders, several processes have been applied such as atomization and milling of solid metals, precipitation from salt metal solutions and electrodeposition [5,6], thermal method [7], microwave irradiation [8], green synthesis [9], chemical reduction [10], and gamma irradiation [11]. Each technique produces a powder with a characteristic morphology that influences its functional properties [12,13].

Nevertheless, in order to achieve better characteristics of silver particles, which affect directly the mechanical and physical properties of the deposited film, and consequently the solar cell performances, the chemical process seems to be the best one giving fine silver powder controlled in shape and size [14]. It was chosen because it did not require special equipment. However, this method involves rigorous processing conditions.

Polyol synthesis was originally introduced by Fievet *et al.* [13] as an excellent method for the production of metallic submicrometer-sized and nano-particles. This method was successfully developed for the preparation of single-crystal silver nano-particles with uniform size and shape using polyvinylpyrrolidone (PVP) [15].

The polyol method involves heating a polyol with a salt precursor and a polymeric capping agent to generate metal colloids. Using this method, silver nano-particles with different shapes have been manufactured [16,17]. All these improvements have promoted the scientific knowledge on nanomaterials [18]. Recently, Rao *et al.* [19] have included strategies which have been employed for the synthesis of nano-materials of different dimensions.

In a typical polyol synthesis, silver atoms are formed by reducing silver nitrate (AgNO_3) used as precursor with ethylene glycol [17]. PVP, a protective agent, plays a crucial part in controlling superfine silver particle size and size distribution by reducing silver nitrate with polyol. The particle size and particle aggregation decrease with an increasing of PVP/ AgNO_3 weight ratio [20,21]. Several works focused on the synthesis of micro- and/or nano-sized silver particles using ethylene glycol [17,22-30].

In this document, we opted for the synthesis of particles using the reduction of silver salt in three gly-

colic media. It must be noted that ethylene glycol was thoroughly employed, however propylene glycol and di-ethylene glycol were rarely used [31,32].

The objective of this work was on one hand to illustrate and assess the difference in the reduction power of three glycols, used in similar concentrations on silver cations, and on other hand to show their influence on the crystallinity and morphological properties of the micro- and nano-particles obtained in the absence and presence of PVP, respectively.

With thusly prepared powders, we have attempted to develop our own purpose paste because most commercial thick-film pastes are not especially developed for solar cell metallization and had to be modified for specific emitters.

EXPERIMENTAL

Materials

Silver samples were prepared through a chemical reduction of a polyol reduced silver nitrate solution using glycol derivatives as solvents. The following commercial reagents were employed: ethylene glycol (EG) from Prolabo, di-ethylene glycol (DEG), PVP and acetone, from Fluka, propylene glycol (PG) from BDH Limited Poole and silver nitrate (AgNO_3) from Sigma-Aldrich, ethyl cellulose from BDH Prolabo, terpineol from Fluka, and polyvinyl- butyral from Sigma Aldrich. All the chemicals were used as received without further purification. De-ionized water was used for washing the glassware.

Preparation of silver micro- and nano-particles

AgNO_3 salt was dissolved under stirring in a polyol reducing agent. Polyols have been tested near their boiling points (197 °C for EG, 244 °C for DEG and, 188 °C for PG). The reaction temperature was fixed approximately between 10 and 20 °C below the polyol boiling point. The molar ratio of the precursor to the polyol was set to 0.1, for each polyol. Each sample batch was divided into two separate samples. One of them was reacted with PVP as a surfactant agent, and the other without PVP. The PVP was weighted in a manner that the weight ratio of AgNO_3 /PVP was 2. For the whole of samples, the precipitation reaction occurs under continuous moderate stirring conditions. At the end of the reactions, the precipitates were separated by centrifugation. They were washed in acetone several times until a clean solvent was obtained. Further drying treatment at 80 °C overnight was performed on the micro-powders obtained without PVP. Colloidal suspensions (nano-particles) with PVP are noted S_{EG} , S_{DEG} and S_{PG} , and the micro-

-powders obtained without PVP are noted P_{EG} , P_{DEG} and P_{PG} . The subscripts EG, DEG and PG correspond to the glycol solvents.

Preparation of conductive pastes

Samples of conductive pastes were formulated using the same kind and amount of glass frit and organic vehicle for comparative purposes with the synthesized silver nano- and micro-powders. They were prepared by dispersing the obtained silver samples and an amount of lead-borosilicate glass frit into an organic-polymeric vehicle (a mixture of ethyl cellulose, terpineol and polyvinyl-butylal) with a mass ratio of 75/5/20 of silver/glass frit/organic vehicle.

Conductive thick films were prepared by following the normal sequence of operations such as paste preparation, screen-printing and firing the pastes. For the back side a commercially aluminium paste from Pemco was used. For the front side, the formulated homemade pastes were screen printed through a stainless steel mesh.

Solar cells fabrication

N+P solar cells were fabricated on as-cut p-doped multicrystalline silicon wafers, 10 cm×10 cm, and about 320 μm in thickness. The achieved process is classic and involves the following steps: *i*) chemical preparation, texturization, *ii*) emitter diffusion, *iii*) anti-reflection coating, *iv*) edge isolation, *v*) back metallization with commercially Ag/Al paste and *vi*) front metallization using the pastes prepared by the synthesized powders.

Conductive thick films were prepared by following the normal sequence of operations such as paste preparation, screen-printing and firing. For the back side, a commercially aluminium paste from Pemco was used. For the front side, the formulated homemade pastes were screenprinted through a stainless steel mesh (mesh count 350). The resultant films for the two sides were dried at ~ 150 °C for 5 min to volatilize the organic binder in the pastes, which otherwise causes gas bubbles at higher temperatures and results in cracking of the metallization [33]. Co-firing of the samples was done in a heated resistance belt furnace with air atmosphere having peak firing temperature of 720 °C for 3 min to sinter the inorganic binder.

The obtained solar cells, hereafter corresponding to P_{EG} , P_{DEG} , P_{PG} and S_{EG} , S_{DEG} , S_{PG} were produced following scrupulously the same process, so that a direct comparison of the results could be made. The pastes were fired on a conveyer belt furnace at a peak set point temperature around 720

°C with roughly 3 s in the hot zone to achieve a thickness of ~ 12 μm .

Analysis methods

The crystalline structure of samples was performed using an X'pert Plus-PANalytical X-ray powder diffractometer using CuK α radiation with a tube current of 50 mA and a voltage of 40 kV. The X'Pert High Score Plus software was used for the phase identification. The study of the morphology was carried out using a scanning electron microscope (Jeol, JSM-6360 LV SEM/EDAX).

Thermal analysis of samples was performed using coupled techniques such as differential scanning calorimeter and thermo-gravimetric analyzer (DSC/TGA). The weight loss of samples was determined by TGA curve. This coupled analysis was performed with a NetzschSTA 409 PC instrument, in the temperature range of 25-1450 °C. This temperature range includes the boiling point of solvents, and degradation temperature of polymers.

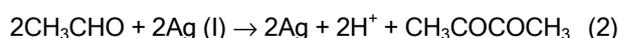
Because silver nano-particles absorb light significantly and their UV/Vis spectra show an intense absorption peak in the visible region, their optical properties were investigated by a Cary 500 DE Varian model, in the spectral range of 175 to 3300 nm. The samples were dispersed in absolute ethanol for the analysis, and the powders were leaved in their final granular form. For the whole, the absorbance measurement was carried out at room temperature, in the spectral range of 300 to 700 nm.

The contact resistance of the deposited layers was measured using standard transmission line measurement (TLM) method. Four contacts were used (2 for current and 2 for voltage).

RESULTS AND DISCUSSION

Chemical reaction description

During the reduction of silver, the transparent mixture changed to yellow, while with continuous stirring the color of the reaction changed to gray and dark gray. These observations show that with an increase in reaction time, concentration of silver cluster gradually increased where silver cations are adsorbed. By analogy and for reason of similarity with other glycols, a general mechanism of metal reduction in ethylene glycol can be represented by the following reactions:



The three silver powders obtained without the use of surfactant have a metallic lustered appearance. However, the samples produced using an excess of PVP are in the form of stable colloidal dispersions of silver nano-particles. All the micro and nano-silver particles synthesized in the same conditions were obtained in a similar time indicating that EG, DEG and PG have identical reduction power. However, EG, DEG and PG have distinct reducibility. In this work the reaction temperature is too high; hence the reaction rates in EG, DEG and PG are accelerated to the same level.

XRD characterization

Figure 1 shows the XRD patterns of the synthesized silver particles in the presence and absence

of PVP. The entire samples exhibit the same patterns. Four strong peaks were noticeable in each pattern, which are characteristics to fcc silver of JCPDS card No. 04-0783 [34]. The results suggest that the powders show good crystallinity. No crystallographic impurities phases were found. The high intensity of the peaks indicated that the silver particles were well crystallized. Nevertheless, the peaks pattern of samples S_{EG} , S_{DEG} and S_{PG} had not changed in crystallinity. These results confirm the negligible amorphous phase of surfactant in PVP samples.

SEM micrographs

The morphologies of silver samples characterized by SEM images are shown in Figure 2 (without PVP for P_{EG} powder) and 3 (with PVP for S_{EG}

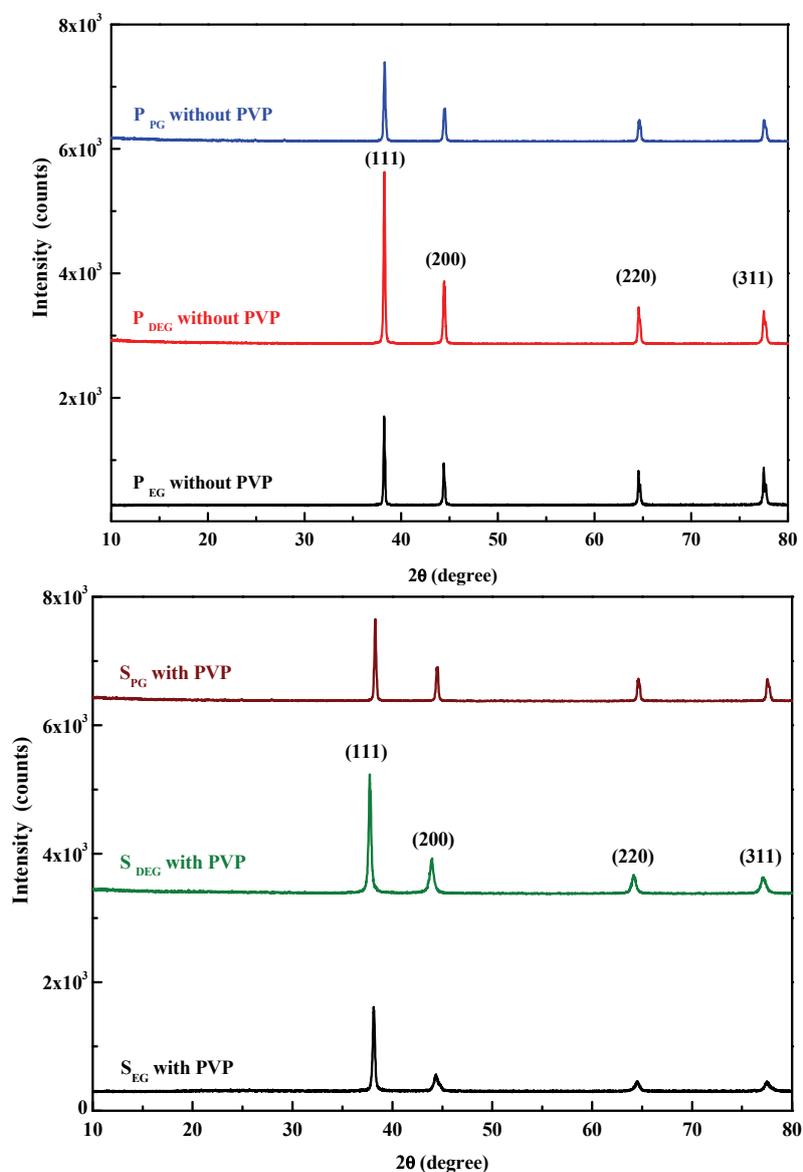


Figure 1. XRD diffraction patterns of the synthesized Ag powders without and with PVP.

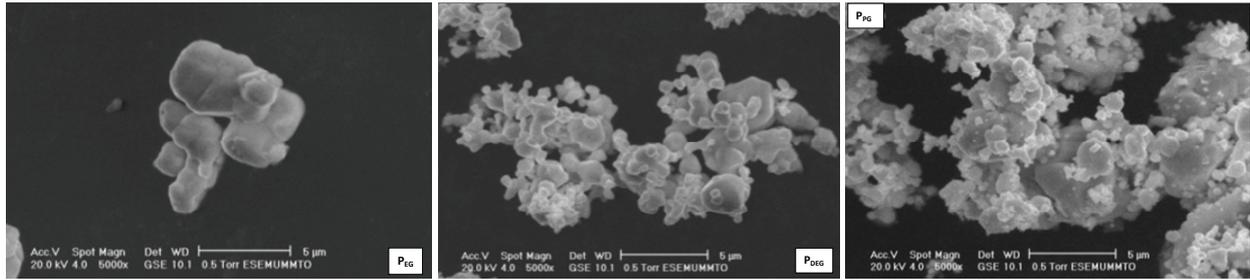


Figure 2. SEM micrographs of the synthesized Ag micro-powder without PVP using respectively EG, DEG and PG.

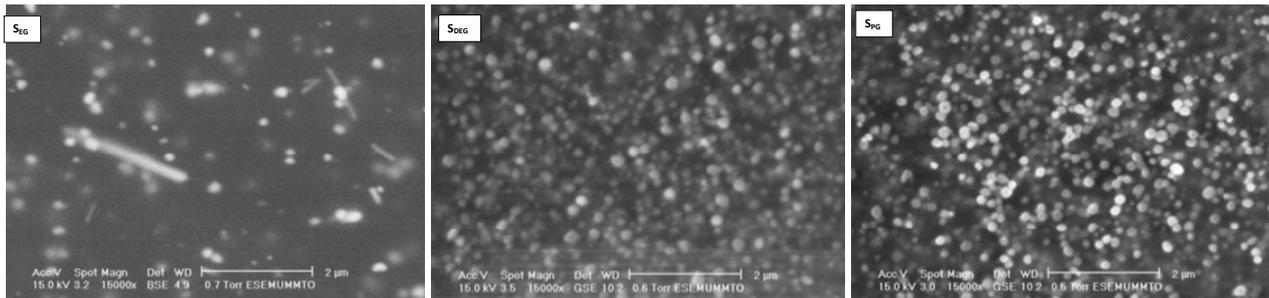


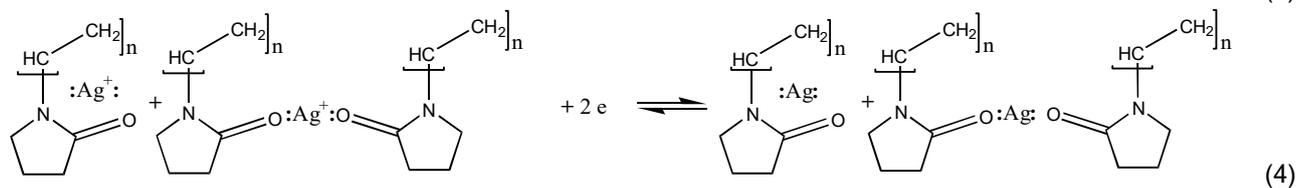
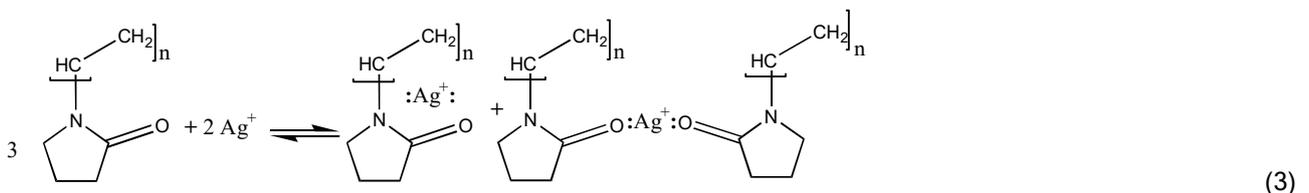
Figure 3. SEM micrographs of the synthesized Ag colloidal suspensions with PVP using respectively EG, DEG and PG.

colloidal suspension). The micrographs revealed that the P_{EG} powder is formed by agglomerated heterogeneous spheroidal particles of size ranging from 1-2 μm. However, the S_{EG} sample prepared with PVP featured homogeneous nano-spherical particles. In addition, the presence of nanowires formed by assembling clusters of some nano-particles was observed. The size was approximately 50-80 nm. The micro-particles of P_{DEG} were agglomerated in a narrow size distribution and their mean size was estimated to be 1-2 μm (Figure 2). The structure of S_{DEG} (Figure 3) was slightly different, as there were no assembled particles forming rods.

The micrograph of P_{PG} presented in Figure 2 illustrates the same morphology for P_{EG} and P_{DEG}. For S_{PG} (Figure 3), spherical silver nano-particles are observed. These results prove that both the size and shape particles depend on the presence of surfactant. These results are in agreement with those obtained by Li *et al.* [35], Yu *et al.* [36] and Gasaymeh *et al.*

[37]. The PVP restricts the mobility of silver ions during the chemical reduction and, prevents aggregation among the particles and limits their sizes. It should be noted that there is no difference in morphological properties of the silver products synthesized in the same conditions.

These effects can be described according to the above described reaction mechanism (Eqs. (3) and (4)). This mechanism is explained by the formation of coordination bonds between PVP and silver ions involving the trapping of Ag⁺ by macromolecules of PVP. Firstly, the complex of PVP silver ions is constructed; secondly, the complex promotes silver nucleation; thirdly, aggregation and limited dispersion leads to the formation of silver nano-particles [38]. As the reaction rate of silver particles increases with the concentration of PVP, it appears that this could be the reason for observing more regular and smaller particles and narrower particle size distributions at higher concentration of PVP [39,40].



Thermal analysis

Figures 4 and 5 show the DSC and TGA curves of P_{EG} and S_{EG} samples from which onset and final decomposition temperatures were obtained. It should be noted that only DSC/TGA data of P_{EG} and S_{EG} were studied because the similarity of the results. From the DSC analysis of silver micro-powders synthesized in EG without PVP (Figure 4), the presence of an exothermic phase transition was observed around 280 °C, while the TGA diagram shows a weight loss of about 0.24%, which is a negligible mass. It represents the amount of organic part that persists as trace of the experimental process. Other impurities located in the micrometer silver powders result from the decomposition of the rest fraction of the reducing agent. As silver does not melt until 961 °C and is not known to sublime, it is reasonable to assume that the observations made during TGA can be attributed to the organic materials only. At the prominent temperatures identified by TGA and DSC, the most important endothermic peak in DSC curve of nano-silver synthesized with PVP, using EG (Figure 5) corresponds to the solvent volatilization of the colloidal suspension. This one coincides with the peaks of weight loss of 18.47 and 20.62%.

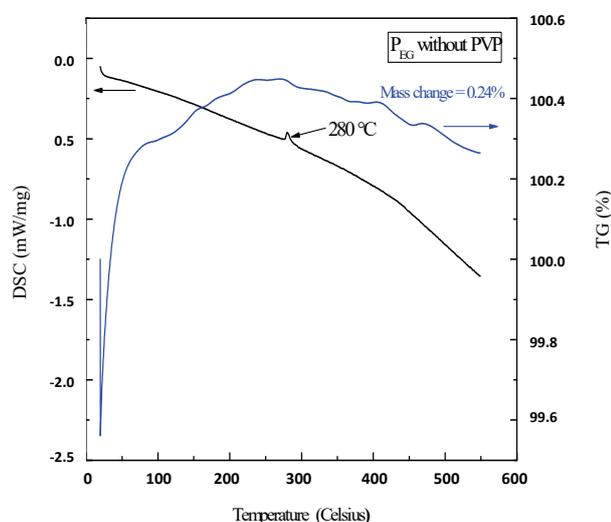


Figure 4. DSC/TGA curves of Ag powders synthesized without PVP using EG.

The organic phase could be however completely removed by keeping the particles at a relatively low temperature (400 °C) for several minutes. In comparison with the results shown in Figure 4, thermal analysis of S_{EG} shows that silver suspensions of nano-particles had a higher degradation temperature than that of PVP used alone. All the nano-silvers present in the colloidal suspensions have almost the

same thermal behavior. Particles coated with stable substances such as PVP are suitable, because the protective layer can be removed at temperatures below 500 °C. This amount of PVP will not affect the conductivity of the metal powder [37,41].

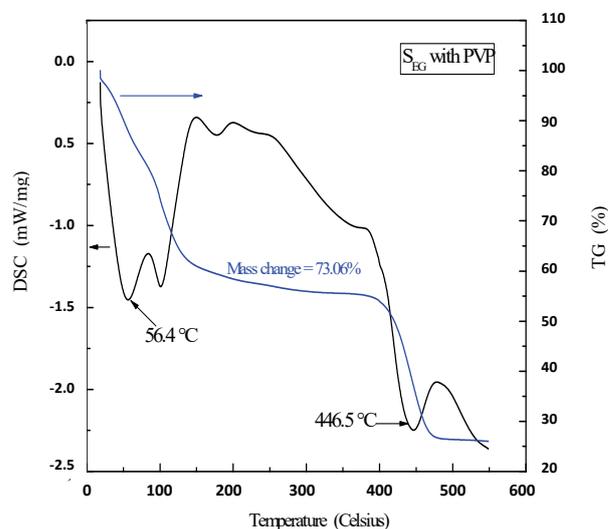


Figure 5. DSC/TGA curves of colloidal nano-suspensions synthesized with PVP using EG.

UV-Vis absorption spectra

Figure 6 shows the UV-Vis absorption spectra for the samples prepared with 0.1 mole ratio of $AgNO_3$ to polyol, in excess of PVP with glycols. The absorption band in visible light region (400–450 nm) is typical for silver nano-particles. The absorbance bands at 447, 441 and 436 nm are attributed to the surface plasmon resonance phenomenon of free electrons in the conduction bands of silver nanoparticle suspensions, respectively synthesized by EG, DEG and PG. These results are in agreement with that of silver nano-particles prepared with PVP chemical reaction by many authors, such as Dung Dang *et al.* [30] and Slistan-Grijalva *et al.* [42]. The morphology and the particle size may play an important role in the absorption spectra of Ag/PVP nano-particles.

Resistivity measurement

The sheet resistance of the fired films for nano-particles was 3.6, 4.1 and $4.6 \times 10^{-6} \Omega \text{ cm}^{-2}$ corresponding to S_{EG} , S_{DEG} and S_{PG} , respectively, and 9.6, 13.6 and $11.1 \times 10^{-6} \Omega \text{ cm}^{-2}$ to P_{EG} , P_{DEG} and P_{PG} , respectively. From these results, we deduce that the conductive behavior of the obtained conductive pastes is dependent on the size, shape and type related to the synthesis process (with or without PVP)

and slightly on the firing range process (around 720 °C) of the screen-printed deposited metallic contacts.

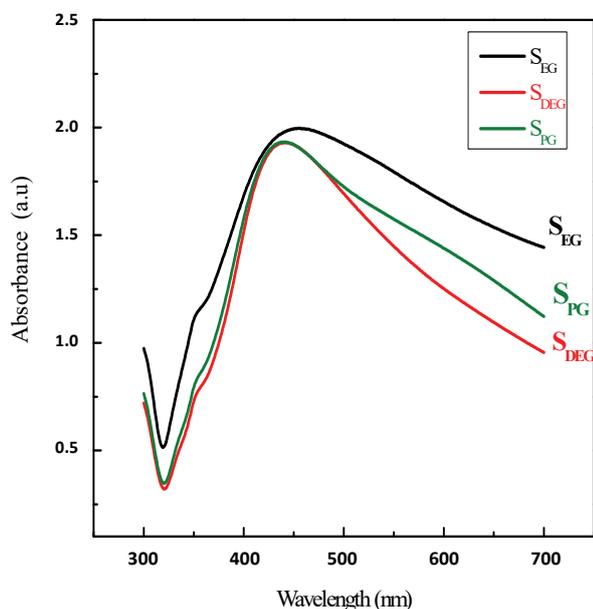


Figure 6. UV/Vis spectra of Ag colloidal nano-suspensions synthesized with PVP.

CONCLUSION

Series of experiments in glycolic medium (EG, DEG and PG) were carried out at the boiling point of each one for the reduction of silver nitrate to silver micro-particles, and to nano-particles with the use of PVP. The three silver powders obtained without the use of surfactant had a metallic lustered appearance. However, the samples produced using an excess of PVP were in the form of stable colloidal dispersions of silver nano-particles.

The obtained results from DRX, SEM and thermal analysis showed that the presence of PVP affects the physicochemical properties of the synthesized silver particles; however, the use of the different glycolic mediums at their boiling temperature has no influence.

The XRD results showed that the powders exhibited good crystallinity, with negligible amorphous phase of surfactant in PVP samples. The SEM of P_{EG} powder reveals that the powder is made by agglomerated heterogeneous spheroidal particles having a size ranging from 1 to 2 μm. However, the S_{EG} sample prepared with PVP exhibits homogeneous nano-spherical particles, with some nanowires formed by assembling clusters of some nano-particles. The size was approximately on the order of 50-80 nm. The micro-particles of P_{DEG} were agglomerated in a narrow size distribution and their mean size was esti-

mated from 1 to 2 μm. The structure of S_{DEG} was slightly different, while the morphologies of P_{EG} and P_{DEG} were comparable. For S_{PG}, spherical silver nano-particles were observed. These results prove that both the size and shape particles depend on the presence of surfactant. The DSC/TGA analysis showed that the protective layer could be removed at temperatures below 500 °C. This amount of PVP will not affect the conductivity of the metal powder. The surface plasmon bands appearing in the UV-visible region are characteristic of silver nano-particles.

The obtained results also show that the highest heating temperature gives similar powders. This means that the heating at high temperature allows the silver nitrate to react completely giving fine powders with same properties.

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NAUČNI RAD

MIKRO- I NANO-ČESTICE SREBRA DOBIJENE POMOĆU RAZLIČITIH GLIKOLA KAO REDUKUJUĆIH SUPSTANCI I MERENJE NJIHOVE PROVODLJIVOSTI

Sinteza mikro i nano-čestica srebra za pripremu provodnih pasti za metalizaciju solarnih ćelija, izvršena je hemijskom redukcijom u prisustvu i odsustvu polivinilpirolidona (PVP). Kao prekursor u sintezi korišćen je srebro nitrat u prisustvu tri poliola (etilen glikol, dietilen glikol i propilen glikol) testiranih na eksperimentalnim temperaturama u blizini njihovih tački ključanja. Na ovaj način je dobijeno šest uzoraka. Bez upotrebe PVP, dobijena su tri uzorka srebra u obliku praha sa metalnim sjajem; međutim, uzorci dobijeni u prisustvu viška PVP se nalaze u formi stabilnih koloidnih disperzija nano-čestica srebra. Strukturna karakterizacija uzoraka pomoću skenirajućeg elektronskog mikroskopa i X-analize pokazala je dobru kristalnost i sfernu morfologiju. DSC i TGA analiza pokazuju da sve nano-čestice srebra prisutne u koloidnoj suspenziji imaju isto termalno ponašanje.

Ključne reči: srebro-nitrat, nano-čestice, hemijska redukcija, derivati glikola, PVP.