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Polyvinyl Alcohol Composite Films Reinforced with Taro (*Colocasia esculenta*) Stem Fiber: Thermal, Mechanical and Biodegradation Studies

Kapil Gulati[a,b], Rachna,[c] Seema Rani[a,d], Mahesh Kumar[e], Geetanjali Jagdeva[a], Suresh Kumar[a], Sohan Lal[a] & Sanjiv Arora[a]

[a]Department of Chemistry, Kurukshetra University, Kurukshetra 136 119, India
[b]Department of Chemistry, Dyal Singh College, Karnal 132 001, India
[c]Department of Chemistry, Ch. Bansi Lal University, Bhiwani 127 021, India
[d]Department of Chemistry, Dr. B.R.A.Govt. College, Kaithal 136 027, India
[e]Department of Chemistry, Mukand Lal National College, Yamunanagar 135 001, India

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Polyvinyl alcohol/Glycerol/Citric acid based composite samples have been synthesized by the method of solvent casting. The composites have been explored in terms of thermal, tensile, biodegradability, water absorption analysis and SEM study with the addition of *Colocasia esculenta* stem fiber (2.5-20.0 %). The equilibrium water absorption percentage with the addition of fiber and upon the increasing content of fiber has been found to be increased with an abrupt change above 12.5 % fiber content. The high value of degradation percentage has been however observed with 20 % loading of fiber (29.13±1.28). Thermal stability of the composite films has also been found to show positive results with the addition of fiber with maximum value of onset degradation temperature obtained with 7.5 % reinforcement. The incorporation of fiber has resulted in enhancement of tensile strength due to modified interfacial adhesion between fiber and matrix with maximum value of 36.06±0.25 MPa obtained for composite with 7.5 % loading of fiber. SEM micrographs have shown the smooth and homogeneous surface of composite film without any loading of fiber (CE-0). High loading of fiber (20 %) however has introduced some voids, holes and cracks in the samples making it easy for water molecules to permeate in the composites responsible for its water absorption percentage.

Keywords: Polyvinyl alchol (PVA), *Colocasia esculenta,* Biodegradability, Mechanical, Thermal

1 Introduction

Growing environmental concerns towards the use of more eco-friendly and sustainable materials by taking into account the new environmental regulations and the social awareness has sparked interest to make use of natural fiber reinforced composites[1,2]. The increasing use of biocomposites has been a promising alternative over synthetic or non-biodegradable petrochemical based plastics[3]. The research on the synthesis of composites using inexpensive and biodegradable polymeric materials has been dynamically pursued[4]. Among the possible options, Polyvinyl alcohol (PVA) is one of the most versatile polymer being water soluble and possessing the several industrial applications with excellent optical and physical properties[5–7]. Nevertheless, certain drawbacks of PVA such as high cost and number of hydroxyl groups limits have been use to serve the purpose.[8] The possible solution to mitigate the problem is the synthesis of polymer composites by using the natural/lignocellulosic fibers as reinforcement to provide the significant properties of resulting composites with excellent biodegradation rate. Lignocellulosic fibers include the examples from both naturally and agricultural industries viz. bamboo, jute, walnut shells, plant leaves or stem fibers etc.[9,10] The key advantages of natural fibers include the superior thermal stability, low density, being renewable, non-toxic and good mechanical properties[11]. In the present work, we have used Taro (*Colocasia esculeneta*) stem powder as reinforcement which is a tropical tubes crop, belongs to the Araceae family.[12] Taro being a fast emerging plant is considered to cause some problems such as harming the water bodies by the thick vegetation in which they colonize and their thick shrub harbor poisonous creatures, pests, insects and breeding place for mosquitoes[13]. Looking on the positive aspects, stems from taro plants are very rich in carbohydrate content and use of the stems as a filler can be also a solution

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*Corresponding author (E-mail: kapil.gulati300@gmail.com)

of the waste utilization. Taro powder filler based HDPE/EVA composites have been synthesized by Hamim et al.[12] In the past few years, extensive efforts have been made to synthesize the composites by using some interfacial or compatibilizing agents to improve the interfacial adhesion between various components which in our case has been made by using the natural organic acid i.e. citric acid. The presence of three carboxyl and one monohydroxyl group has made its use to serve as a cross-linker[14]. Due to its multi carboxyl groups, esterification reaction can be easily setup between citric acid and hydroxyl groups of PVA and/or Taro stem powder. In the present research, the method of solvent casting has been adopted to synthesize PVA/Taro (*Colocasia esculenta*) biocomposites with various loadings of fiber by making use of glycerol as a plasticizer for an enhancement in the flexibility of the samples. The resulting composites have been characterized in terms of thermal, mechanical, water absorption, biodegradation and morphological studies**.**

2 Materials and Methods

2.1 Materials

Taro (*Colocasia esculenta*) leaves were picked from its plant (Kurukshetra University, Kurukshetra). Polyvinyl alcohol (PVA) (Mw = 85,000-1,24,000, degree of hydrolysis = 86.0-89.0 %) was supplied by SDFCL (s d fine-chem limited). Glycerol and citric acid were supplied by Himedia (India). Deionised water was used for the preparation of composite films.

2.2 Methodology

Synthesis of polymer composite thin films of polyvinyl alcohol (PVA) based matrix, reinforced with *colocasia esculenta* stem fiber (CE) was carried out by solvent casting method, with different fiber loadings of 2.5, 5, 7.5, 10, 12.5, 15 and 20% of PVA (Table 1).

The stems of *colocasia esculenta* plant were dried under sunlight and powdered. The obtained fiber was sieved under 18 mesh size sieve. Firstly, PVA was added in hot deionised water at 70-80 °C. The solution was magnetically stirred for about half an hour to dissolve it completely. The saturated solution of glycerol (plasticizer) and aqueous solution of citric acid was added to the beaker containing PVA solution. Then, it was followed by the addition of homogenised solution of CE fiber in water. The solution was kept under stirring continuously for 3 h at 50 °C. The resultant mixture was sonicated in Ultra cleaner sonicator for 15 min. The solution was allowed to cool at room temperature. Films were cast into clean and dry glass dishes of 15cm x 5cm. Finally all the films were dried in vacuum oven at 65 °C for about 40 h to make certain the complete exclusion of engrossed moisture and kept in airtight polyethyelene bags until further investigation.

Table 1 — Different compositions of the PVA, Glycerol, Citric acid and CE fiber

Sample Index	PVA (g)	Glycerol (g)	Citric acid (g)	Fiber content (%/g)
CE-0	5	2.5	0.05	0
CE-1	5	2.5	0.05	2.5/0.125
CE-2	5	2.5	0.05	5.0/0.250
CE-3	5	2.5	0.05	7.5/0.375
CE-4	5	2.5	0.05	10.0/0.50
CE-5	5	2.5	0.05	12.5/0.625
CE-6	5	2.5	0.05	15.0/0.750

2.3 Characterizations

2.3.1 FTIR spectra

FTIR spectra were recorded on a MB-3000 ABB spectrophotometer in transmission mode over the frequency range of 4000-600 cm^{-1} to characterize the presence of any functional group in composite films.

2.3.2 Water absorption study

This method was adopted to calculate the weight change of composites immersed in water. The film cuts (30 mm x 30 mm) dried to the constant weight (w_i) were submerged in water for 24 h at room temperature which then were taken out and the final weight of the absorbed films was recorded (w_f) after removing the water left at surface by absorbing it with filter paper. There were three samples of each composition and the average readings were taken. The water absorption (%) of each film was determined by using the following formula:

$$\text{Water absorption (\%)} = \frac{wf-wi}{wi} \times 100$$

2.3.3 Soil burial degradability

This method was used to determine the biodegradability of specimens which were buried in the soil. The degradation of the samples was observed by measuring the weight change of the samples withdrawn from the soil on every 20 days for two months.

2.3.4 Thermogravimetric analysis (TGA)

TGA of the samples was carried out on Thermal gravimetric Analyzer at the heating rate of 10 °C min^{-1} under the nitrogen atmosphere from ambient temperature to 600 °C.

2.3.5 Mechanical Properties

Tensile testing of samples was carried out on Universal Testing Machine (UTM) AG-IS Shimadzu, Galaxy Plywood Industries, Yamuna Nagar Haryana in accordance with ASTM-D882 standards. The cross head speed was maintained at 10 mm/min.

2.3.6 SEM Analysis

SEM analysis of samples was performed on JEOL JSM-6510LV to observe the morphology of composite films. Films were coated with gold particles to prevent any damages by the electron beam. Samples were scanned at 1000x magnification and 15 kV power.

2.3.7 Statistical Analysis

Statistical analysis of composites was performed with individually prepared samples in triplicate, as the replicated results are provided with mean ± standard deviation (SD) values. Analysis of variance (ANOVA) was conducted and the significance of each mean property value was estimated ($P < 0.05$).

3 Results and Discussion

3.1 FTIR Spectroscopy

The Fig. 1 (a & b) depicts the FTIR spectra of PVA/Glycerol/Citric acid based polymer composite film (CE-0) and *Colocasia esculenta* reinforced composite film with fiber loading of 7.5 % (CE-3) respectively. The broad high absorption band at ~3300 cm^{-1} is assumed to arise from –OH stretching frequency of polyvinyl alcohol and absorbed moisture.

Where CE-0= PVA/Glycerol/Citric acid and CE-3= PVA/Glycerol/Citric acid/ 7.5 % stem fiber.

The characteristic band due to the presence of –CH stretching vibrations of long chains of alkanes are shown at 2923 cm^{-1}. The peak located at around 2854 cm^{-1} is attributed to the asymmetric stretching of –CH group of matrix. The band obtained at 2358 cm^{-1} is attributed to the presence of atmospheric carbon dioxide. The vibrational band at 1724 cm^{-1} is related to the stretching vibrational peak of carbonyl group obtained due to the incomplete hydrolysis of polyvinyl acetate during the synthesis of PVA[15] or arises due to the presence of ester bonds and carboxyl group in citric acid[16]. The band located at 1087 cm^{-1} is related to the –C–O stretching of PVA. The absorption band at 1457 and 1440 cm^{-1} were assigned to the bending vibration of –CH2 and –CH3 deformation[17]. In the characteristic spectra of CE-3, the peaks obtained at 1240 cm^{-1} represents the medium strong vibrations of –C–O group due to the connection of matrix and filler.[12] Also, a decrease in the intensity of vibrational bands arising from the bending vibrations of =C-H and =CH2 of alkanes indicates the bonding formation between polymer matrix and reinforcement in the presence of citric acid as cross linking agent[12].

3.2 Water Absorption

Where CE-0= Polyvinyl alcohol and glycerol composite film without any loading of fiber and CE-1, CE-2, CE-3, CE-4, CE-5, CE-6, CE-7 are Polyvinyl alcohol composites with 2.5, 5.0, 7.5, 10.0, 12.5, 15.0 and 20.0 % loading of *Colocaisa esculenta* stem fiber respectively.

The water absorption percentage of polyvinyl alcohol composites reinforced with various loadings of fiber is presented in Fig. 2. The equilibrium water

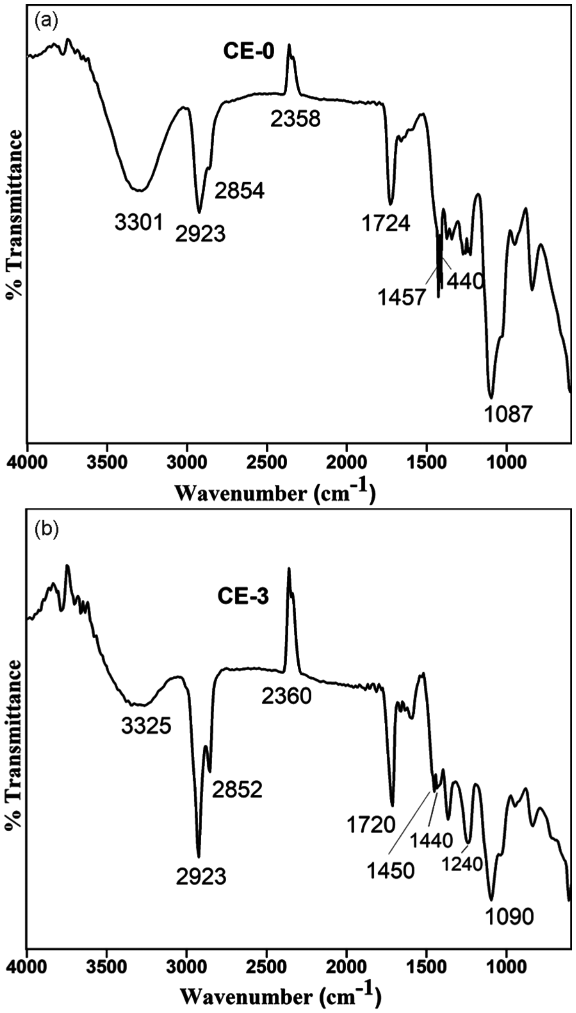

Fig. 1 — FTIR Spectra of (a) CE-0, and (b) CE-3.

absorption percentage with the addition of fiber and upon the increasing content of fiber was found to be increased. The reason for this increasing percentage might be due to the existence of hydrophilic lignocellulosic fiber in composites which caused the formation of hydrogen bonding between the free hydroxyl groups of fiber and water molecules present. The results were in complete agreement with the data observed by Hamim et al.[12] and Shih et al.[18] There was an abrupt change of absorption percentage above 12.5 to 20 % loading of fiber in comparison to CE-0 (60.88±0.88). The significant increase of water absorption percentage at high fiber loading is clearly visible in Figure 2. The high loading of *Colocasia esculenta* fiber enhanced the filler-filler interaction which caused the decrease in adhesion between fiber and polymer matrix henceforth made it more susceptible to the absorption of water during the time of immersion. The results of water absorption data were subjected to the analysis of variance by one way ANOVA at the significance level of 0.05 (Table 2). By interpretation of data obtained from ANOVA, it was found that different loading of fiber has a significant effect on the water absorption percentage as confirmed from the value of probability factor of the order of 3.93042 x 10^{-10}.

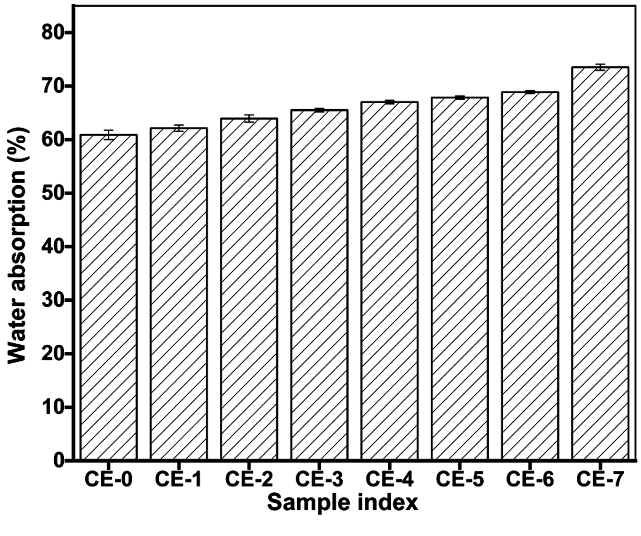

Fig. 2 — Water absorption of PVA composites with different fiber loading.

3.3 Biodegradability

The percentage of weight loss of polymer composites is shown in Fig. 3. Samples with same dimensions were chosen to minimize the effect of films shape on the percentage of biodegradability. The weight loss percentage was determined at the regular interval of 20 days for 2 months. The composite films did not show any significant change in the percentage of weight loss in the initial 20 days. However, at the end of 60 days the virgin composite sample of PVA/Glycerol/Citric acid (CE-0) showed the maximum resistance towards degradation with weight loss percentage of (18.46±0.4) at the end of 60 days. The composite samples from 2.5 to 7.5 % reinforcement of fiber (CE-1 to CE-3) showed almost similar and least degradation percentage among the other samples incorporated with any amount of fiber. The composite sample with 20 % loading of fiber (CE-7) at the end of 60 days showed the maximum degradation percentage (29.13±1.28) of weight loss. The pattern of degradation percentage can be explained on the basis of degree of cross linking obtained as shown in the SEM micrographs also.

Where CE-0= Polyvinyl alcohol and glycerol composite film without any loading of fiber and CE-1, CE-2, CE-3, CE-4, CE-5, CE-6, CE-7 are Polyvinyl alcohol composites with 2.5, 5.0, 7.5, 10.0, 12.5, 15.0 and 20.0 % loading of *Colocaisa esculenta* stem fiber respectively.

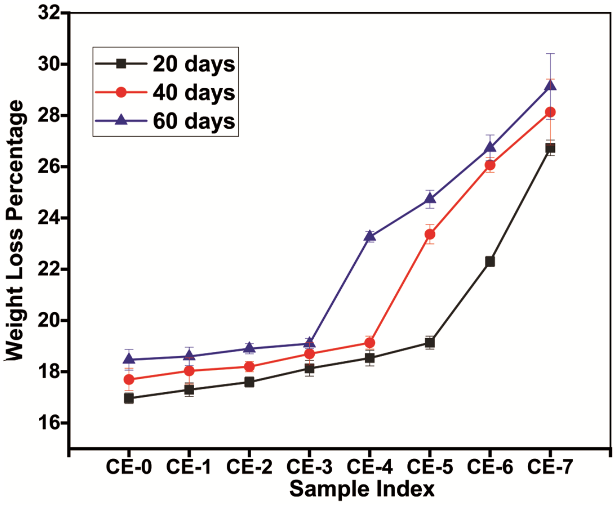

Fig. 3 — Weight loss percentage of composite films.

Table 2 — One way ANOVA on water absorption data

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Composition	7	344.12975	49.16139	57.08144	3.93042E-10
Error	16	13.78	0.86125	--	--
Total	23	357.90975	--	--	--

As highly cross linked films with good adhesion resist the degradation and shows the lower degradation rate[19].. The higher degradation percentage in composite with 20 % reinforcement (CE-7) can be explained on the basis of its high water absorption capacity (Fig. 3) which was responsible for making it more prone to the attack of microorganisms.[20]

Also, the presence of free volume, cracks and voids in composite at high fiber loading of 20 % reinforcement (CE-7) was responsible for its high weight loss percentage. The data of weight loss percentage was subjected to Two way ANOVA. It was found that at significance level of 0.05 there were considerable differences from one level of composition to another (Table 3). Also, significant results were obtained in respect of time of degradation on degradability data.

3.4 Thermal Study

3.4.1 Thermaogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) curves of Polyvinyl alcohol/Glycerol/Citric acid composite films with various loadings of *Colocasia esculenta* fiber are exhibited in Fig. 4 while Table 4 and Table 5 shows the degradation temperature range, temperature at which 50 % weight loss occurred (T_{50}) and char residue (%). From both the informations, it was found that PVA composites filled with various loadings of fiber showed the onset degradation temperature towards higher side (Table 4). Also, the residual percentage was found to be increased with reinforcement with maximum value obtained for PVA composites with 7.5 % fiber content (8.68 %) in comparison to virgin sample (3.75 %).

Where, CE-0= Polyvinyl alcohol and glycerol composite film without any loading of fiber and CE-2, CE-3, CE-4, CE-6, CE-7 are Polyvinyl alcohol composites with 5.0, 7.5, 10.0, 15.0 and 20.0 % loading of *Colocaisa esculenta* stem fiber respectively.

From the data elaborated, PVA composites with fiber reinforcement exhibited the high thermal stability than neat sample. This was observed due to reinforcement of lignocellulosic fiber with major constituent of cellulose in which it is expected the thermal degradation of fiber above 400 °C[21]. The major component of fiber i.e. cellulose sustained the physical properties of natural fiber and had a significant contribution in the thermal stability of composite films[22]. High thermal stability of

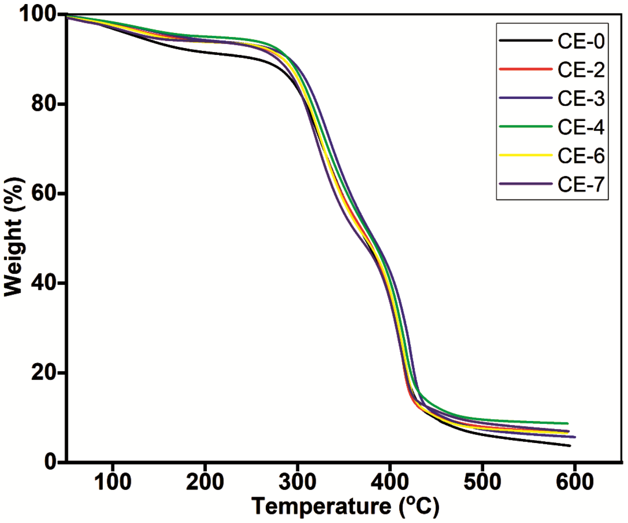

Fig. 4 — Thermogravimetric analysis of PVA composites at the heating rate of 10 °C min^{-1}.

Table 3 — Two way ANOVA on biodegradability data

Source	DF	Sum of Squares	Mean Square	F Value	P Value
Composition	7	907.86875	129.69554	554.84721	1.17885E-43
Degradation time period	2	93.26028	46.63014	199.48723	5.53356E-24
Interaction	14	50.62417	3.61601	15.46957	3.04866E-13
Composition	23	1051.75319	45.7284	195.62952	7.74942E-40
Error	48	11.22	0.23375	--	--
Corrected Total	71	1062.97319	--	--	--

Table 4 — Characteristic thermal decomposition data of composite films

Sample index	First degradation stage				Second degradation stage			
	T_o (°C)	T_{max} (°C)	T_{end} (°C)	MWLR (%min^{-1})	T_o (°C)	T_{max} (°C)	T_{end} (°C)	MWLR (%min^{-1})
CE-0	291.8	319.6	341.7	1.11	390.9	414.7	450.9	1.72
CE-2	291.5	321.8	351.3	1.24	391.2	416.1	441.3	1.87
CE-3	295.2	332.2	365.2	1.34	394.4	422.9	444.2	2.10
CE-4	280.3	311.7	360.5	1.49	394.9	417.9	444.2	2.23
CE-6	279.2	326.7	359.6	1.54	385.3	415.5	435.1	2.34
CE-7	276.4	318.7	346.1	5.61	382.7	411.1	432.1	6.52

composites was attributed to the degradation of *Colocasia esculenta* fiber which built a barrier to the gas exhaust from thermoplastic matrix throughout the process of heating.

Where, CE-0= Polyvinyl alcohol and glycerol composite film without any loading of fiber and CE-2, CE-3, CE-4, CE-6, CE-7 are Polyvinyl alcohol composites with 5.0, 7.5, 10.0, 15.0 and 20.0 % loading of *Colocaisa esculenta* stem fiber respectively. T_o, T_{max} and T_{end} stands for onset, maximum degradation and endset temperature respectively. MWLR stands for maximum weight loss rate.

Other parameter in supporting factor of increasing thermal stability was temperature at which 50% weight loss of composites occurred (T_{50}) which was found to be increased with reinforcement up to 7.5% (Table 5). However at further loading, there was a decrease in thermal degradation temperature and hence thermal stability. This can be explained on the basis of non homogeneous dispersion of fiber in matrix which resulted into weak adhesion among fiber and matrix; led to reduction in thermal stability of resulting composites.

3.5 Tensile Properties

Figure 5 demonstrates the tensile properties of Polyvinyl alcohol (PVA) composites with different fiber loading. From this figure, a significant enhancement of tensile strength can be seen in PVA composites with the addition of fiber compared to the raw polymer composite. When the fiber content increased to 7.5% (CE-3), the tensile strength increased to its maximum value of 36.06±0.25 MPa. The incorporation of fiber resulted in enhancement of tensile strength owing to the modified interfacial fixing within fiber and matrix. The active groups from cross linking agent citric acid interacted with hydroxyl groups of *Colocasia esculenta* fiber forming hydrogen bonding within the composites. Thus, the composites with low fiber content involved more in hydrogen bonding and hence were with the presence of lesser number of free hydroxyl groups and in doing so, showed low hydrophilic character in comparison to composites with high reinforcement (Fig. 2). After 7.5% loading, tensile strength decreased progressively due to the poor shifting of stress transfer between matrix and fiber by means of weak interfacial adhesion.

Table 5 — Temperature of 50 % weight loss and residual mass of PVA composites

Sample index	T_{50} (°C)	Char Residue (%)
CE-0	372.84	3.75
CE-2	373.05	6.62
CE-3	382.05	8.68
CE-4	380.02	6.95
CE-6	373.58	6.57
CE-7	367.91	5.68

Where, CE-0= Polyvinyl alcohol and glycerol composite film without any loading of fiber and CE-1, CE-2, CE-3, CE-4, CE-5, CE-6, CE-7 are Polyvinyl alcohol composites with 2.5, 5.0, 7.5, 10.0, 12.5, 15.0 and 20.0% loading of *Colocaisa esculenta* stem fiber respectively.

This behaviour was observed due to the agglomeration of fiber at higher content and hence contributed to the poor stress transfer and thus creating voids in the samples on the application of external stress. Young's modulus of PVA composites as a function of filler content (sample index) is also shown in the Fig. 5 (a).

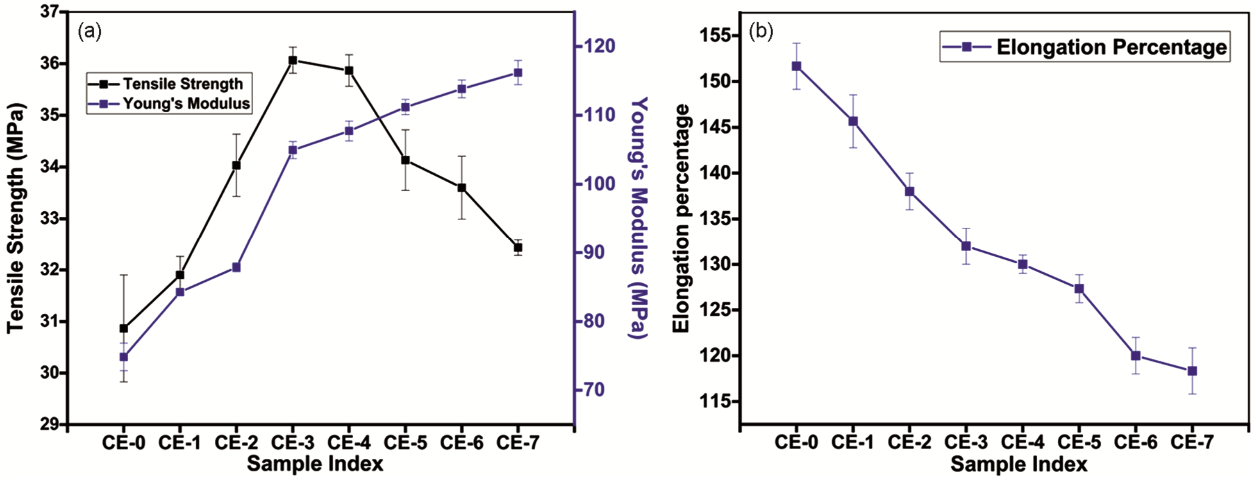

Fig. 5 — (a) Tensile strength, Young's modulus, and (b) Elongation percentage of PVA composites.

A continuous improvement was observed in Young's modulus of composites with the addition of filler and was high compared to the neat blend with maximum value displayed by composites loaded with 20 % fiber content. The increase in Young's modulus with the addition of fiber was predicted due to the presence of rigid filler content which offered the polymer composites with significant stiffness characteristics responsible for the brittleness of the composites. An introduction of small/rigid fiber particles also provide additional strengthening in structures improving the modulus of elasticity by enhancing the total surface area exchange in the interface between fiber and matrix. The same conclusion was also reported by Zhao et al.[23] with the addition of sisal fiber in HDPE matrix.

All the data of tensile properties were subjected to one way ANOVA at significance level ($p<0.05$) showing considerable differences in the properties on the addition of fiber (Table 6 and Table 7). Addition of fiber reduced the flexibility of polymer matrix responsible for making the PVA based composites more rigid and hence decreased the elongation percentage significantly (Table 8).

3.6 Morphology Analysis

SEM images of the surface of composite films at different magnifications are shown in Fig. 6. The pure PVA/Glycerol/citric acid composite without any loading of fiber (CE-0) displayed a smooth and homogeneous surface without the presence of any cracks/pores (Fig. 6 a). Compared with the surface of CE-0, the cross-section of PVA film with 7.5% addition of fiber (CE-3) was compact and highly cross linked with somewhat roughed surface due to the presence of fiber in matrix (Fig. 6 c). No distinct

Table 6 — One way ANOVA on Tensile strength data

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Composition	7	70.3012	10.04303	32.68284	2.54819E-8
Error	16	4.9166	0.30729	--	--
Total	23	75.2178	--	--	--

Table 7 — One way ANOVA on Young's modulus data

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Composition	7	5075.18625	725.02661	407.31832	8.24693E-17
Error	16	28.48	1.78	--	--
Total	23	5103.66625	--	--	--

Table 8 — One way ANOVA on Elongation percentage data

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Composition	7	2879.95833	411.42262	90.58847	1.13093E-11
Error	16	72.66667	4.54167	--	--
Total	23	2952.625	--	--	--

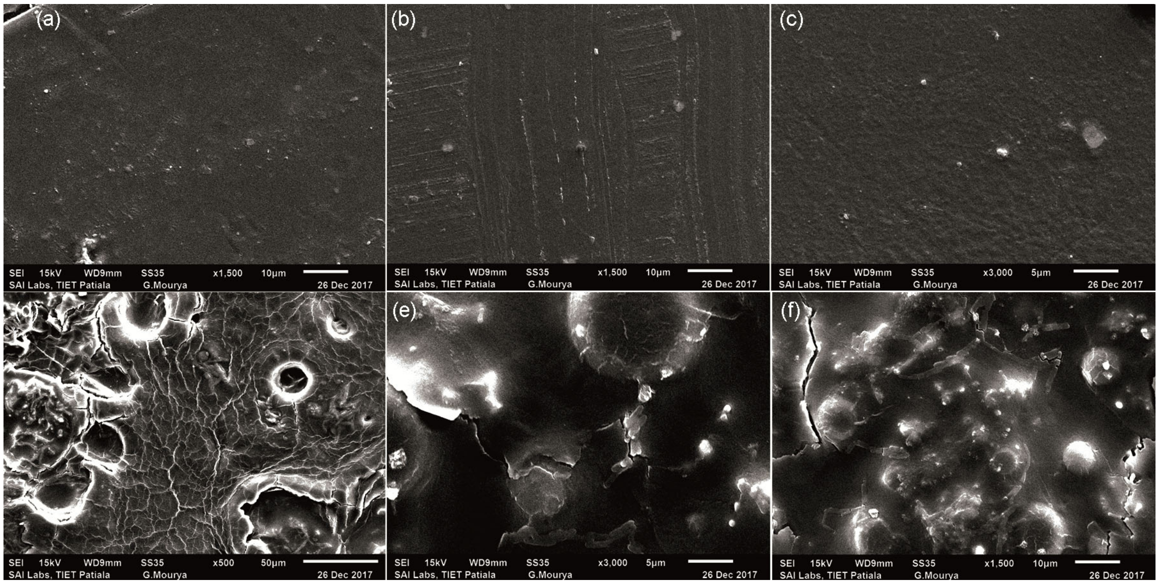

Fig. 6 — SEM micrographs of PVA composites at different magnifications.

separation of polymer matrix was however seen with the addition of fiber, showing the compatibility of the composite formed between fiber and matrix. On the other hand, the high loading of fiber led to the change in the surface structure with the presence of voids, holes and cracks (Fig. 6 (d) & (e)). The presence of hollow spaces also made it easy for water molecules to permeate in the composites responsible for high water uptaking (Fig. 2) during water absorption testing. Thus, it was observed that high loading of *Colocasia esculenta* (fiber) was not susceptible by PVA matrix leading to the inhomogeneous dispersion of fiber in the matrix. It was concluded that Polyvinyl alcohol/Glycerol/Citric acid based composites with low content of *Colocasia esculenta* fiber, were highly cross linked and compact with the optimum results observed for 7.5 % loading of fiber in terms of thermal, tensile and SEM analysis.

Where CE-0= Polyvinyl alcohol and glycerol composite film without any loading of fiber and CE-3 and CE-7 are Polyvinyl alcohol composites with 7.5 and 20.0 % loading of *Colocaisa esculenta* stem fiber respectively.

4 Conclusion

Polyvinyl alcohol/Glycerol/Citric acid based composite samples have been synthesized by the method of solvent casting. It is concluded that PVA based composites with low content of *Colocasia esculenta* fiber, are highly cross linked and compact; with the optimum results observed for 7.5% loading of fiber in terms of thermal, tensile and SEM analysis.

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