Mechanical Performance of Nanoclay-Cellulose Fibre Particulate Composites Fabricated by Modified Two Stage Wet/Hand Layup Method

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Abstract Polymer composites are multiphase materials, where the reinforcement in the form the short/long fibre, filler, particulate enveloped by a matrix. In case of particulate composites, different shapes and sizes of it are dispersed into matrix in random manner. This class of materials is introduced to cheapen the price of polymer and achieve specific behavior may be improved mechanical performance, electrical resistance etc. Firstly cellulose fibre, nanoclay is analyzed for its particle size and size of particles is also found from Scanning Electron Micrograph image to get the clear, rough idea about particulates respectively. An experimental effort is made to manufacture cellulose-nanoclay, nanoclay reinforced vinyl ester composites lamina by Modified Two Stage Hand/Wet Layup Method (MTSHLM) and the specimens for various tests viz. tensile, flexural and bending are machined according to ASTM standards. This innovative method of making composites are experimentally ensured the presence of entire particulates i.e. cellulose, nanoclay. Further the dimension of the specimen made in stage 1, in thickness direction, along its length is measured using micrometer to understand the capability of lamina manufactured by MTSHLM. After characterizing of composites under mechanical load revealed that cellulose acts as a supplement in cellulose-nanoclay composites. An increase in nanoclay content in nanoclay composites is also result in reasonably good improvement of mechanical properties, particularly under bending load. The highest tensile strength, modulus of 32.65, 383.55 MPa is achieved in 5 % cellulose fibre—6.67 % nanoclay composition. In three-point-bend test the composition contains 5 % Cellulose fibre—6.67 % nanoclay lead to 104.13 MPa, 3.73 GPa flexural strength, modulus respectively. But the composition of 6.67 % nanoclay vinyl ester composites has exhibited Charpy impact strength of 2.17 kJ/m^2. Tested composite specimens are analyzed under scanning electron microscope to know dispersion of particulate, interfacial bond between reinforcement and matrix.

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1 Introduction

One of the earthy materials occurring in the nature is clay and possesses plasticity when it is wet. At this stage clay can be molded to any form and is retained after drying (Ries 1906). When the molded items/articles are heated, the material becomes very hard e.g. brick(s) used in construction, drinking water stored in pots.

The main uses of the clay have been recognized since the earliest periods of civilization. Many references are available in the ancient Assyrian and Egyptians records to the employment of clay for manufacture of bricks and for fulling or whitening cloth (Searle 1912).

A careful observations is made and review conducted to understand the manufacturing method of composites, introduction of reinforcements and role i.e. cellulose, nanoclay on different matrices like bio, thermoplastic, thermoset. Further mechanical performance of composites is understood from the past works systematically. Microcrystalline cellulose powder of different concentrations was partially dissolved in lithium chloride/N, N-dimethylacetamide to produce cast nanocomposite film. Then by using Zwick 20 kN Universal Testing Machine tensile tests was conducted on samples at a cross head displacement rate of 1 mm/min to determine their strength, modulus (Gindl and Keckes 2005). Bionanocomposites films were prepared with the combination of poly lactic acid as matrix and microfibrillated cellulose as reinforcement through solvent evaporation at room temperature (Tingaut et al. 2010). Fully green composites were laminated and pressed at 190 °C from polylactic acid, bamboo fibre, microfibrillated cellulose. The specimens of 40 × 3 × 1.5 mm were tested at a cross head speed of 2 mm/min to determine bending properties (Okubo et al. 2005).

Composite films made form micro to nano fibrillated cellulose and polyurethane were subjected to tensile load for knowing strength, modulus (Ozgur and Seydibeyoglu 2008). Atactic polyvinyl alcohol solution contains different concentrations of microfibrillated cellulose is sonicated (1, 5 min) and stirred for 24 h. Then the films (100 μm) were cast on to glass plate with controlled leveling and dried at room temperature for 7 days to determine tensile properties using miniature materials tester Minimat at a cross head speed of 5 mm/min (Jue et al. 2008). With the help of Instron 3365 universal materials tester tensile test was conducted on specimens of 38 × 4.5 × 0.35 mm made from films to find young's modulus, strength and strain at fracture (Nakagaito and Yano 2008).

High Density Poly Ethylene (HDPE, grade HD6605), pine flour and organic clay were blended in thermo kinetic high-shear mixer i.e. K-mixer at 5000 rpm and is discharged at 190 °C. Then the granulated blends were injection molded through

Vista Sentry VSX at 199 °C and a mold temperature of 100 °C to determine tensile, flexural and impact performance (Lei et al. 2007). Melt, direct dry blending processes were adopted to introduce nanoclay into HDPE to make wood-plastic, wood-flour composites by compounding. Tensile, flexural tests were carried out using an Instron 5585 H at a test head speed of 50, 1.3 mm/min respectively (Faruk and Matuana Laurent 2008).

Using Barbender mixing machine chopped wood, coir fibre (2 mm length), poly propylene granules, montmorillonite nanoclay were heated in Hot Press Machine at 170 °C to make hybrid composites for tensile test (Islam et al. 2015). Medium Density Fibreboard (MDF) was manufactured using aspen fibre, Urea Formaldehyde resin, monmorillonite type nanoclay at different contents to test their mechanical performance (Ashori and Nourbakhsh 2009).

Two different batches of composites films were made using Poly-L-Lactide, Montmorillonite clay (Cloisite 25 A) by extrusion. Then tensile test was conducted at a speed of 10 mm/min on Instron 4481 mechanical tester (Lewitus et al. 2006). Poly Vinyl Chloride, hectorite, bentonite clay based composites were made and tensile tested (Awad Walid et al. 2009). Modified soy protein concentrate (SPC) resin was prepared by blending SPC with nano-clay particles, then cross linked with glutaraldehyde. This matrix was combined with flax yarn, fabric to prepare composites and find their tensile, flexural properties (Huang and Netravali 2007).

The effect of montmorillonite nanoclay reinforcement in epoxy resin was investigated through flexural test (Zainuddin et al. 2010). Bisphenol A diglycidyl ether (D.E.R. 332), Nanocor Organophilic clay mixture was ultrasonically stirred using JAC ultrasonic 1505 bath for 1, 3 h to find the dispersion effect by Dynamic Mechanical Test (Wang and Qin 2007). Ultrasound sonication effect in nanoclay clusters of nanoclay/araldite epoxy composites was investigated (Lam et al. 2005). Nanoclay was firstly introduced into epoxy resin and is manually mixed with glass rod before it was mechanically stirred at 500 rpm for 1 h. The mixture was heated in a hot water bath at 50 °C to reduce viscosity of resin and then the hardener is introduced. Using MTS machine alliance RT/50 tensile test was conducted at a cross head speed of 2 mm/min on dog bone shaped composites (Ho et al. 2006). With the aid of mechanical shearing nanocomposites were prepared by in situ polymerization method. The composites were tested for tensile strain, strength and modulus (Qi et al. 2006).

From the above and literature review of about 2000 papers from various journals, it is understood that Two Stage Hand/Wet Layup was firstly introduced, which ensures the complete presence of dora hemp particulate fibre into polyester resin. Then the composites were mechanically tested (Srinivasababu 2015).

In the current work an attempt is made to reinforce nanoclay alone, cellulose-nanoclay into vinyl ester matrix and the composites mechanical performance is also evaluated along with the analysis of particulates i.e. cellulose, nanoclay. The terms cellulose fibre and cellulose are used invariably in this chapter.

2 Materials, Manufacturing and Testing

The materials used in this work are vinyl ester resin, cellulose, nanoclay, procured from Ecmas Resins Pvt. Ltd., Hyderabad, Telangana and Sigma-Aldrich Corporation Bangalore, India respectively.

2.1 Matrix—Vinyl Ester Resin

Ecmalon 9911 is bisphenol epoxy based vinyl ester resin is used as matrix. This kind of resins is suitable for the fabrication of chemical equipment because they offer good resistance to degradation in contact with variety of chemicals which include strong acid and alkalies. Paper and pulp industry use Bisphenol epoxy vinyl ester resins. This resin is suited for wet layup, pultrusion and other types of molding. The properties of the liquid resin are tested according to IS6746-1994 by the manufacturer, are given in Table 1.

Shelf life of resin is 3 months from the date of manufacture when it is stored at temperature below 25 °C. The resin shelf life drops significantly at higher temperatures. The specimens were made from the cast sheets cured 24, 4 h at room temperature and 90 °C respectively. Mechanical properties of the tested specimens were provided by Ecmas Resins, given in Table 2.

Table 1 Properties of Ecmalon 9913 vinyl ester resin (provided by manufacturer)

Property	Description/value
Appearance	Clear, yellow liquid
Viscosity @25 °C (cps)	400 (Brookfield viscometer)
Specific gravity (25/25 °C)	1.05
Acid value (mg KOH/g)	10
Volatiles @150 °C (%)	42
Gel time @25 °C	25 min

Table 2 Mechanical properties of Ecmalon 9913 vinyl ester resin cast specimens (provided by manufacturer)

Property	Unit	Typical value	Test method
Tensile strength	Mpa	70	ISO 527
Tensile modulus	Mpa	3200	ISO 527
Elongation at break	%	4.0	ISO 527
Flexural strength	Mpa	115	ISO 178
Flexural modulus	Mpa	3000	ISO 178
Heat deflection temp	°C	102	ISO 75
Hardness	Barcol	35	IS6746-'94

Table 3 Specification of cellulose medium fibre (provided by manufacturer)

Test	Specification
Appearance (color)	White to off white
Appearance (form)	Powder or solid
Moisture content	≤10 %

Table 4 Specification of nanoclay (provided by manufacturer)

Test	Specification
Appearance (color)	White to off-white
Appearance (form)	Powder
Loss on drying	3.0 %
Size	≤20 μm
Density (bulk density)	200–500 kg/m^3

2.2 Reinforcements—Cellulose Fibre, Nanoclay

One of the reinforcement used in this work is cellulose fibers (CF). Sigma Aldrich has specified the fiber and is given in Table 3. The applications of cellulose fibres are for partition chromatography. Cellulose (medium fibers) is also used in metabolic pathway and carbohydrate applications. It has been used to study biofuel and biorefinery applications. The relative density of fibre is 0.6 gm/cm^3. Surface modified nanoclay (Montmorillonite clay) contains 0.5 wt% aminipropyltriethoxysilane, 15–35 wt% octadecylamine and the properties provided by supplier is given in Table 4. This nanoclay (NC) was recommended by manufacturer for melt compounding with polypropylene (PP), polyethylene (PE), and ethylene vinyl acetate (EVA) and provided following properties when it was reinforced.

- Improve modulus
- Increase barrier
- Enhance chemical resistance
- Improve flame retardancy
- Increase heat deflection temperature (HDT)

2.3 Manufacturing

In this experimentation two batches of specimens are fabricated by using Two Stage Wet/Hand Layup manufacturing method published elsewhere (Srinivasababu 2015) but with some modification i.e. MTSHLM and are described subsequently.

Initially vinyl ester resin is poured in 1000 ml capacity Borosil beaker and the nanoclay is introduced into it slowly by stirring with a glass rod of 6 in. length. Once the resin-nanoclay is mixed manually, it is further stirred in a mechanical

Table 5 Composition of cellulose fibre, nanoclay

Composition label in graph	Composition label	Cellulose fibre content (%)	Nanoclay content (%)
A	A1	5	3.33
B	B1	5	6.67
C	C1	5	10
A	A2	–	3.33
B	B2	–	6.67
C	C2	–	10

mixer up to 5 min. Now the mixture is poured through a Borosil beaker with 10 mm hole into the mold till it is completely occupied on entire surface and allowed to cure at room temperature up to 30 min. Once the resin-cellulose mixture in partially soft solid form, resin is poured into the mold again till complete thickness is attained as per the dimensions specified by ASTM standards and is allowed to cure at room temperature for 24 h. During preparation of lamina promoter, accelerator, catalyst 2 % of each is mixed in the resin one after another in stage-1 lamina preparation. But the percentage of the above chemicals is 2.2 in stage-2 lamina preparations to enhance the bond between the two layers.

Secondly, the next batch of specimens is prepared with the following additional steps. Cellulose fibre, nanoclay are introduced into vinyl ester matrix one after another by ensuring thorough mixing of the reinforcements in resin. The compositions of CF, NC used in this experimentation is given in Table 5.

The prepared lamina is heated in NSW-143 Oven Universal up to 5 h and allowed to cool at room temperature and is shown in Fig. 1. Using mechanically operated cut machine tensile, flexural and impact test specimens are obtained from lamina as per the specifications of ASTM D638-10, ASTM D 790-07 and ASTM D6110-08 for tensile, flexural and Charpy impact test respectively. With the help of belt grinding machine the specimens are ground to ensure flatness and finishing of edges.

2.4 Testing

Cellulose, nanoclay particle size is analyzed using Particle Size Analyser at Kelvin Labs, Hyderabad, Telangana, India. Further reinforcement samples are characterized for its morphology and size using Scanning Electron Microscope.

Tensile, flexural tests are conducted using PC 2000 Electronic Tensometer at a crosshead speed of 2, 8 mm/min respectively. A notch is cut to a depth of 2.5 mm using Notch Cutter and Charpy impact test is conducted on Computerized Izod/Charpy impact tester. In all the mechanical tests five specimens are tested in each case and the average value is taken.

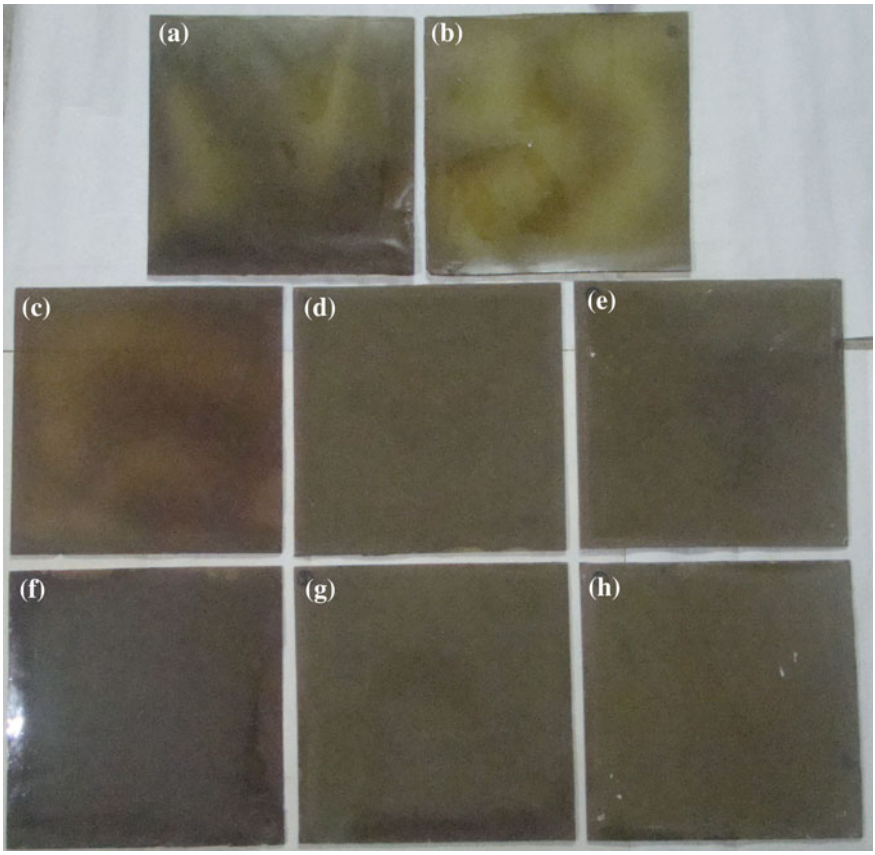

Fig. 1 **a**, **b** Vinyl ester Plain, **c** 3.33 wt% NC, **d** 6.67 wt% NC, **e** 10 wt% NC, **f** 5 wt% CF-3.33 wt% NC, **g** 5 wt% CF-6.67 wt% NC, **h** 5 wt% CF-10 wt% NC Composite lamina manufactured by Two Stage Hand/Wet Layup Manufacturing Method

3 Results and Discussion

The reinforcement viz. cellulose fibre, nanoclay are analyzed for size, morphology and two batches of composites are tested for understanding their manufacturing ability, mechanical performance.

3.1 Cellulose Fibre, Nanoclay—Size, Morphology

The nanoclay, cellulose fibre is tested for its size, run 1, 2, 3 and average is reported in Figs. 2a–d and 3a–d respectively. In case of nanoclay the peak diameter varied

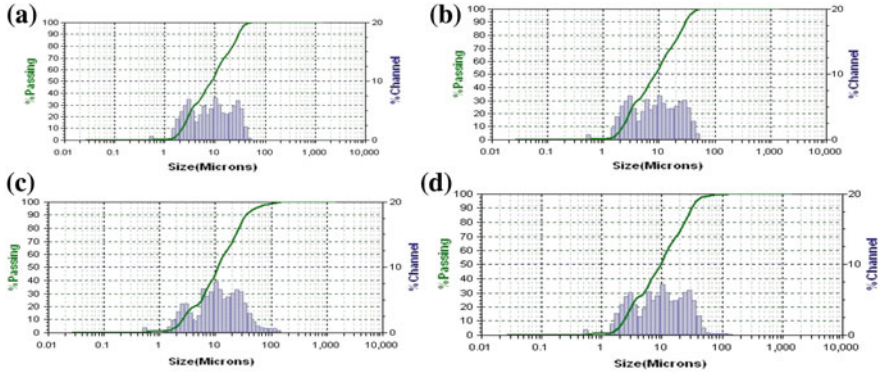

Fig. 2 Particle size analysis of Nanoclay **a** Run 1, **b** Run 2, **c** Run 3, **d** Average

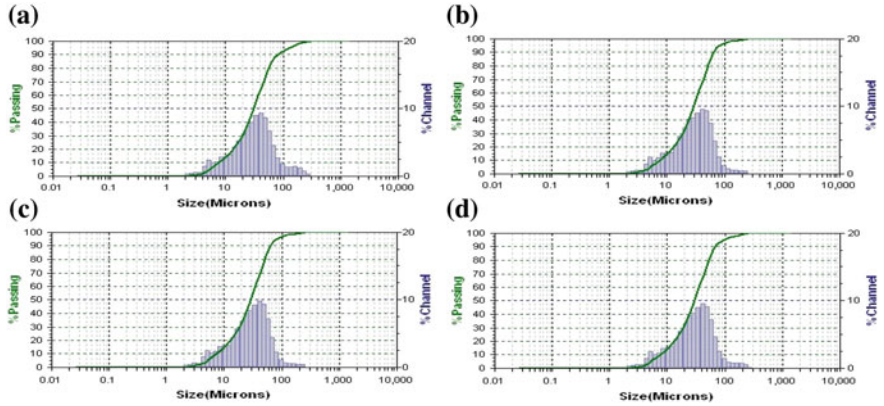

Fig. 3 Particle size analysis of Cellulose Fibre **a** Run 1, **b** Run 2, **c** Run 3, **d** Average

from 2.531 to 24.55 nm whereas 50 percentile particles are of 9.45 µm. Formation of clusters with variable size is clearly visible from Scanning Electron Micrograph (SEM), Fig. 4. Now the size of more/less big size clusters is measured and its value is 154–229 µm, the shape of the particle is nearly spherical. Formation of soft heap solid mass is recognized from Fig. 5 i.e. cellulose fibre SEM. In this case fibre group size varies as 119–230 µm. Due to micron size of the cellulose fibre it seems to be spherical in its shape.

3.2 Mechanical Properties

In this work plain, CF-NC, NC vinyl ester composite specimens prepared from lamina are tested as per ASTM standards and the average value of five specimens is

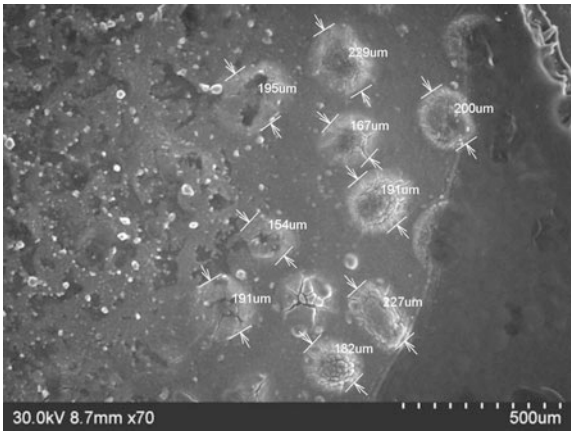

Fig. 4 Scanning Electron Microscope image of Nanoclay with particle size

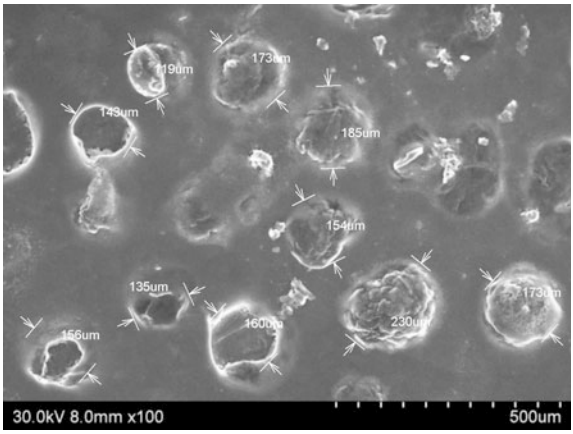

Fig. 5 Scanning Electron Microscope image of contains particle size, geometry of Cellulose Fibre

calculated in each test. The tensile strength of the CF-NC, NC vinyl ester composites is decreased with increase in NC content due to the lack of sufficient matrix. The reinforcement effect of CF is clearly recognizable from Fig. 6, where the tensile strength of CF-NC composites at A1, B1, C1 compositions is greater than A2, B2, C2 respectively. The highest value of 32.65 MPa is achieved at composition A1. Elongation is clearly decreased with increase in NC content at all compositions and moreover the composition reinforced with CF have more elongation when compared to others composites possess no CF. Tensile modulus of the composites against composition is shown in Fig. 7. Highest tensile modulus (383.55 MPa) in tension is obtained for the composites at A1 composition.

In all the composites tensile strength, modulus is varied with respect to composition. An expected reasonable amount of properties enhancement is not observed due to disparity in thickness of lamina prepared in stage-1 of the process along its length, width. With an eye observation it is clear that the lamina prepared

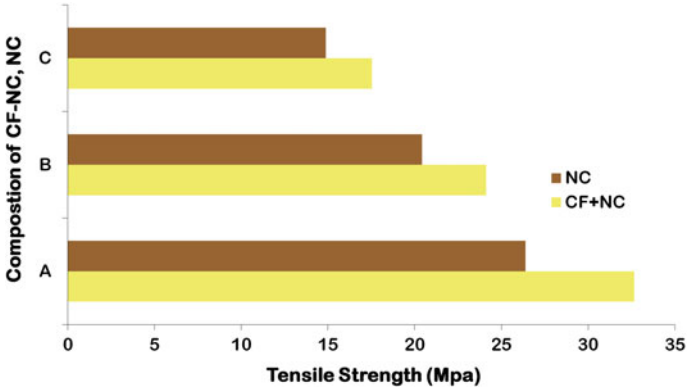

Fig. 6 Tensile strength of CF-NC, NC reinforced vinyl ester composites at different compositions

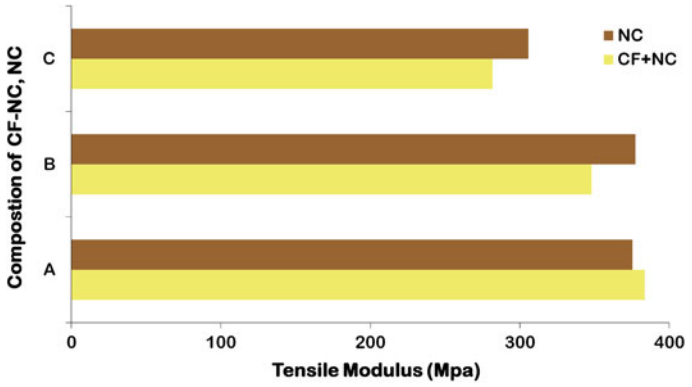

Fig. 7 Tensile modulus of CF-NC, NC reinforced vinyl ester composites at different compositions

at highest content of NC unable to spread over the mold surface due to high viscosity possessed by the resin-reinforcement mixture. Hence thickness of the specimens manufactured in stage-1 is measured at five different locations along its length and is given in Table 6 for CF-NC, NC composites.

Miniaturized tensile test was conducted (Ozgur and Seydibeyoglu 2008) on different compositions of polyurethane and cellulose fibre composites, where polyurethane and cellulose are hydrophilic. Poly urethane-cellulose nano fibre1 has exhibited maximum tensile strength.

Flexural strength, modulus of the CF-NC, NC composites is increased with NC content except at its initial, highest level. At initial level the reinforcement acts as filler, high level lack of matrix in stage-1. Figure 8 and 9 shows the flexural strength, modulus at different composition of CF/NC, NC contents. The highest flexural strength of 104.13 MPa is attained for the composition B1. This tells that

Table 6 Thickness of tensile test specimens manufactured in stage-1

Composition A1: 5 % CF + 3.33 % NC					
Specimen number	Thickness (mm)				
	Position 1	Position 2	Position 3	Position 4	Position 5
1	3.169	3.192	3.566	3.461	3.180
2	2.559	3.028	3.408	3.387	3.305
3	3.040	3.437	3.791	3.157	2.772
4	2.763	2.304	2.516	2.953	3.253
5	2.947	3.326	3.115	3.087	3.331
Composition B1: 5 % CF + 6.67 % NC					
6	4.588	3.370	3.479	3.208	2.255
7	4.910	4.198	3.831	3.700	3.550
8	4.196	3.774	3.120	2.715	3.448
9	2.949	2.550	2.551	3.817	3.97
10	2.570	3.113	3.493	3.873	2.733
Composition C1: 5 % CF + 10 % NC					
11	2.205	3.281	3.154	1.901	2.802
12	3.542	2.351	2.385	2.466	2.288
13	2.898	1.929	1.878	1.928	2.813
14	3.005	3.152	2.388	2.432	2.402
15	3.381	2.020	2.706	2.143	2.675
Composition A2: 3.33 % NC					
16	2.755	2.909	3.528	3.420	2.591
17	3.497	2.798	2.685	2.476	2.279
18	4.016	3.638	3.088	3.320	2.998
19	2.989	3.068	2.607	3.927	3.682
20	2.254	2.488	2.971	3.311	3.385
Composition B2: 6.67 % NC					
21	3.240	2.698	2.608	2.560	2.579
22	3.286	2.899	2.762	2.762	2.358
23	3.030	2.929	2.885	2.626	2.942
24	2.926	2.935	2.891	2.630	2.721
25	2.604	2.461	2.693	2.545	2.405
Composition C2: 10 % NC					
26	2.233	1.593	3.247	3.281	3.292
27	3.435	2.207	3.130	3.112	2.898
28	2.530	3.126	2.196	2.397	2.894
29	3.507	2.998	1.506	2.117	1.798
30	2.569	3.259	3.879	3.878	3.044

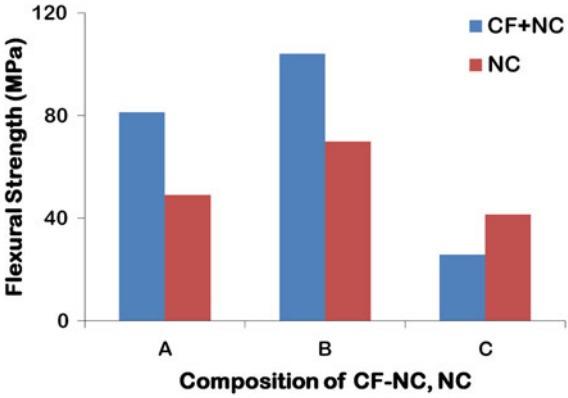

Fig. 8 Flexural strength of CF-NC, NC reinforced vinyl ester composites at different compositions

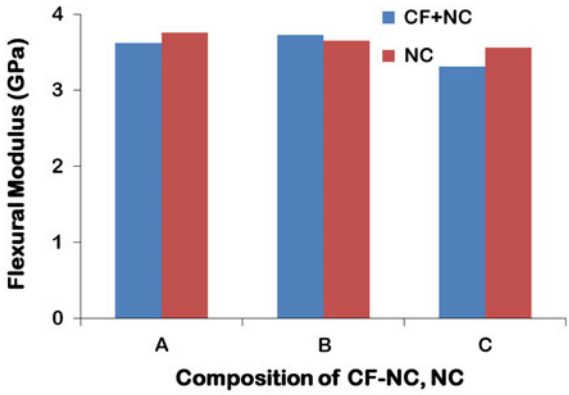

Fig. 9 Flexural modulus of CF-NC, NC reinforced vinyl ester composites at different compositions

increase in NC has enhanced the flexural strength of composites and bears load. All composites are failed due to bending only, is identified after the test. NC at highest compositions i.e. C1, C2 thickness of first stage process itself occupied maximum mold and thereby second stage process of manufacturing has very minimum thickness results in decrease in flexural strength, modulus of the CF/NC, NC composites. Interestingly the flexural strength at C2 is 37.62 % more when compares with composites have composition C1. The highest flexural modulus 3.73, 3.76 is achieved by the compositions B1, A1 composites respectively. Thickness of flexural test specimens is given in Table 7 which is taken at five different sites.

Impact strength of the CF-NC, NC reinforced vinyl ester composites are increased with its content(s) except at C1, C2 compositions due to insufficient presence of the matrix. The highest Chrapy impact strength of 1.9, 2.17 kJ/m^2 is achieved for the composites with the compositions B1, B2 and is visible from Fig. 10. All the composites have exhibited 'C' type failure as per ASTM D6110-08. In these test specimens the thickness is measured at five different positions and is tabulated in Table 8.

Table 7 Thickness of flexural test specimens manufactured in stage-1

Composition A1: 5 % CF + 3.33 % NC					
Specimen number	Thickness (mm)				
	Position 1	Position 2	Position 3	Position 4	Position 5
1	2.598	2.448	2.385	2.313	2.643
2	2.673	2.598	2.519	2.513	2.428
3	3.046	2.962	2.496	2.493	2.448
4	3.307	2.791	3.683	2.709	2.818
5	3.106	3.216	3.259	4.14	2.875
Composition B1: 5 % CF + 6.67 % NC					
1	4.553	4.557	4.394	2.995	2.415
2	2.72	2.38	2.806	2.672	2.681
3	3.324	3.543	3.711	4.136	4.13
4	4.278	4.266	4.285	3.754	3.405
5	4.56	4.344	3.881	3.737	2.977
Composition C1: 5 % CF + 10 % NC					
1	3.14	4.465	2.241	2.604	3.965
2	4.167	3.735	2.974	3.644	4.536
3	3.535	2.923	3.389	2.672	3.903
4	3.193	2.962	2.822	2.964	3.328
5	2.053	3.342	3.183	2.073	1.929
Composition A2: 3.33 % NC					
1	3.546	2.936	2.596	2.762	3.579
2	3.45	3.639	3.89	3.416	3.886
3	4.193	3.883	3.978	4.052	4.409
4	3.579	3.285	3.141	2.632	2.118
5	4.896	4.618	4.381	4.09	3.643
Composition B2: 6.67 % NC					
1	3.112	2.796	2.492	2.875	2.834
2	3.006	2.939	2.819	2.409	3.114
3	3.298	3.396	3.313	3.487	3.81
4	3.072	3.163	2.84	3.018	3.03
5	3.63	3.435	3.282	3.155	3.207
Composition C2: 10 % NC					
1	2.905	3.339	2.245	1.992	2.445
2	2.564	2.686	2.178	3.151	2.791
3	3.935	4.267	3.403	3.144	3.031
4	3.106	3.564	3.381	1.958	2.713
5	3.43	2.61	3.41	3.569	3.65

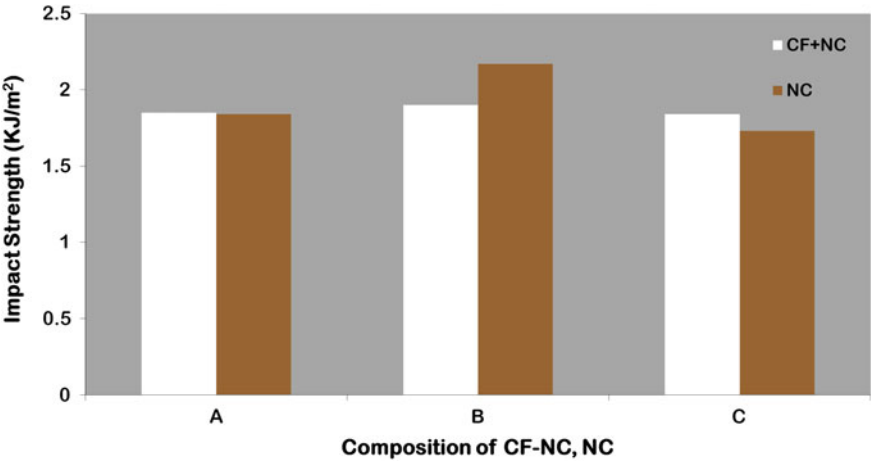

Fig. 10 Charpy impact strength of CF-NC, NC reinforced vinyl ester composites at different compositions

Table 8 Thickness of Charpy impact test specimens manufactured in stage-1

Specimen number	Thickness (mm)				
	Position 1	Position 2	Position 3	Position 4	Position 5
Composition A1: 5 % CF + 3.33 % NC					
1	2.488	2.652	2.648	2.478	2.38
2	3.059	3.101	3.778	2.241	2.229
3	2.465	2.747	2.438	2.368	2.662
4	2.955	2.681	2.786	2.725	2.596
5	2.63	3.077	3.046	2.926	3.657
Composition B1: 5 % CF + 6.67 % NC					
6	2.867	2.141	2.37	2.155	2.001
7	1.966	2.654	2.565	2.484	2.447
8	2.776	2.204	2.469	2.571	1.964
9	2.728	3.073	3.102	3.812	4.637
10	2.766	2.578	2.611	2.451	2.08
Composition C1: 5 % CF + 10 % NC					
11	2.772	4.518	3.79	4.02	3.798
12	2.517	2.667	2.68	2.246	1.579
13	3.176	3.01	2.789	2.861	2.861
14	3.307	2.294	3.695	2.861	1.929
15	2.207	2.551	2.819	1.888	2.278

(continued)

Table 8 (continued)

Specimen number	Thickness (mm)				
	Position 1	Position 2	Position 3	Position 4	Position 5
Composition A2: 3.33 NC					
16	3.652	4.208	4.23	4.12	4.308
17	4.314	3.331	2.23	2.707	3.114
18	3.73	2.989	3.399	3.75	3.776
19	3.136	3.046	3.01	3.932	3.28
20	4.166	4.12	3.96	3.794	3.859
Composition B2: 6.67 % NC					
21	4.309	4.296	4.209	3.814	4.036
22	4.1	4.019	3.608	3.766	3.893
23	3.991	3.627	3.621	3.549	2.881
24	3.974	3.91	3.784	3.361	2.644
25	3.374	3.369	3.313	4.378	4.427
Composition C2: 10 % NC					
26	3.612	3.973	3.864	2.372	2.012
27	2.914	2.643	2.262	3.072	2.349
28	2.926	3.678	3.065	2.547	2.797
29	3.768	3.96	2.418	3.202	2.433
30	2.101	2.071	2.601	3.949	4.716

3.3 Morphology Study

After testing the composites at different load viz. tensile, flexural and Charpy impact, the fractured specimen surface is examined under Scanning Electron Microscope and the following observations are made. In performing morphology study overall view, four different locations are observed in order to understand the dispersion, distribution of reinforcements and bond between them and vinyl ester matrix.

The presence of white lines (Fig. 11a) over entire surface of the sample given belief that the nanoclay presence along with cellulose. Figure 11a–d tells that the agglomerated particulates are comes peel out from the matrix, whose size varies. A rougher fracture surface is identified from the surface of the specimen, visible in Fig. 11b–e.

Two layers (stage-1: composite; stage-2: plain matrix) in the composite lamina are clearly distinguishable from Fig. 12 a, b. A rougher and smooth fractured

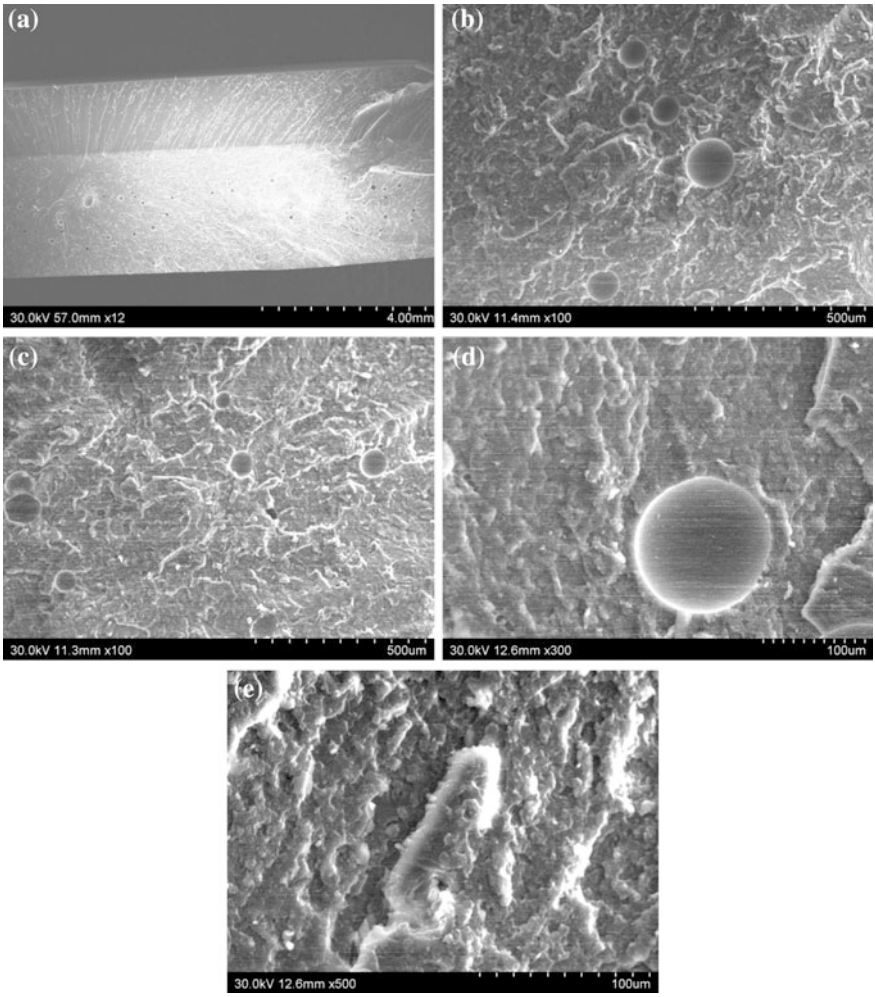

Fig. 11 SEM image of tensile tested 5 % CF-3.33 % NC reinforced vinyl ester composites **a** overall view, **b** location 1, **c** location 2, **c** location 3, **d** location 4, **e** location 5

surface and some amount agglomerates are observed in Fig. 12b, c respectively. Narrow cleavage is visible from Fig. 12d, e.

Specimens are failed at its outer most layer due initiation of crack in three-point-bend test and identified from the SEM image of Fig. 13a, which contains smooth, rough fractured surfaces along with the voids that indicates the presence of particulates before test. Rougher surface, diversified dimples with

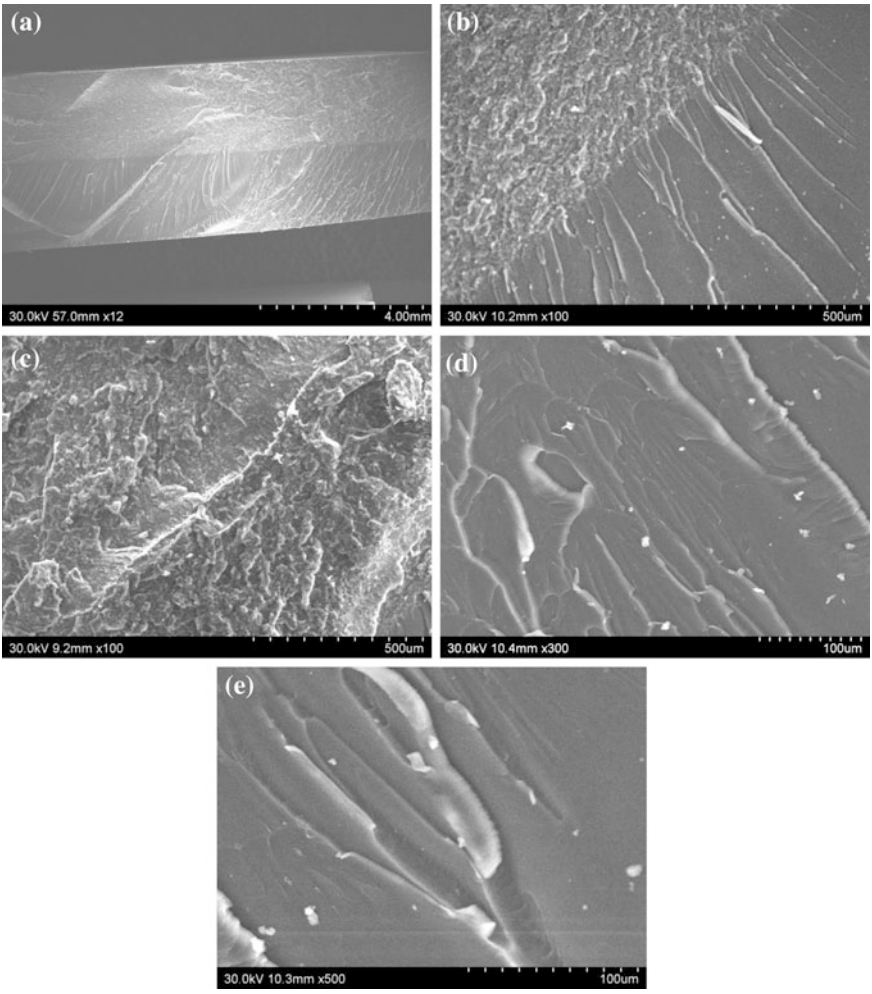

Fig. 12 SEM image of tensile tested 3.33 % NC reinforced vinyl ester composites **a** overall view, **b** location 1, **c** location 2, **c** location 3, **d** location 4, **e** location 5

circular projected agglomerates are visible from Fig. 13b–e. Nearly spherical shaped voids are identified from the fractured surfaces of nanoclay composites, Fig. 14a–e.

SEM image of fractured surface of the impact tested CF-NC, NC reinforced vinyl ester composites are shown in Figs. 15a–e and 16a–e respectively. A rougher uneven surface, even surface is observed in Figs. 15 and 16 respectively.

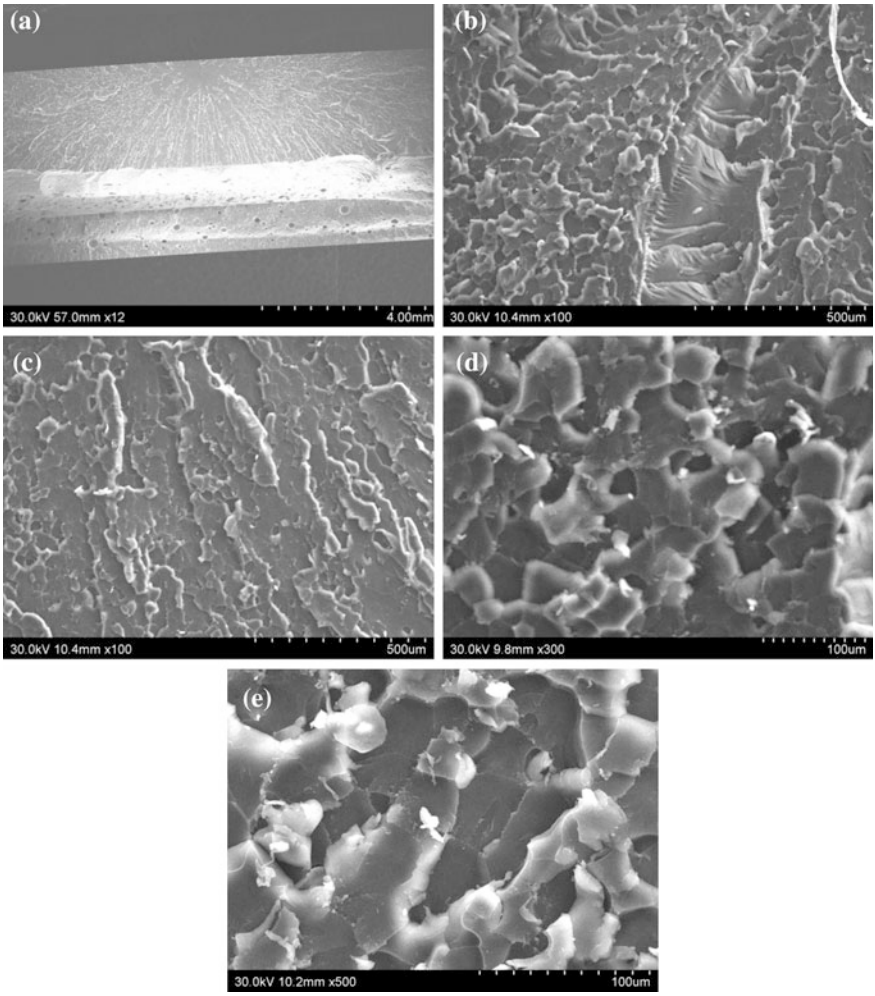

Fig. 13 SEM image of flexural tested 5 % CF-6.67 % NC reinforced vinyl ester composites **a** overall view, **b** location 1, **c** location 2, **c** location 3, **d** location 4, **e** location 5

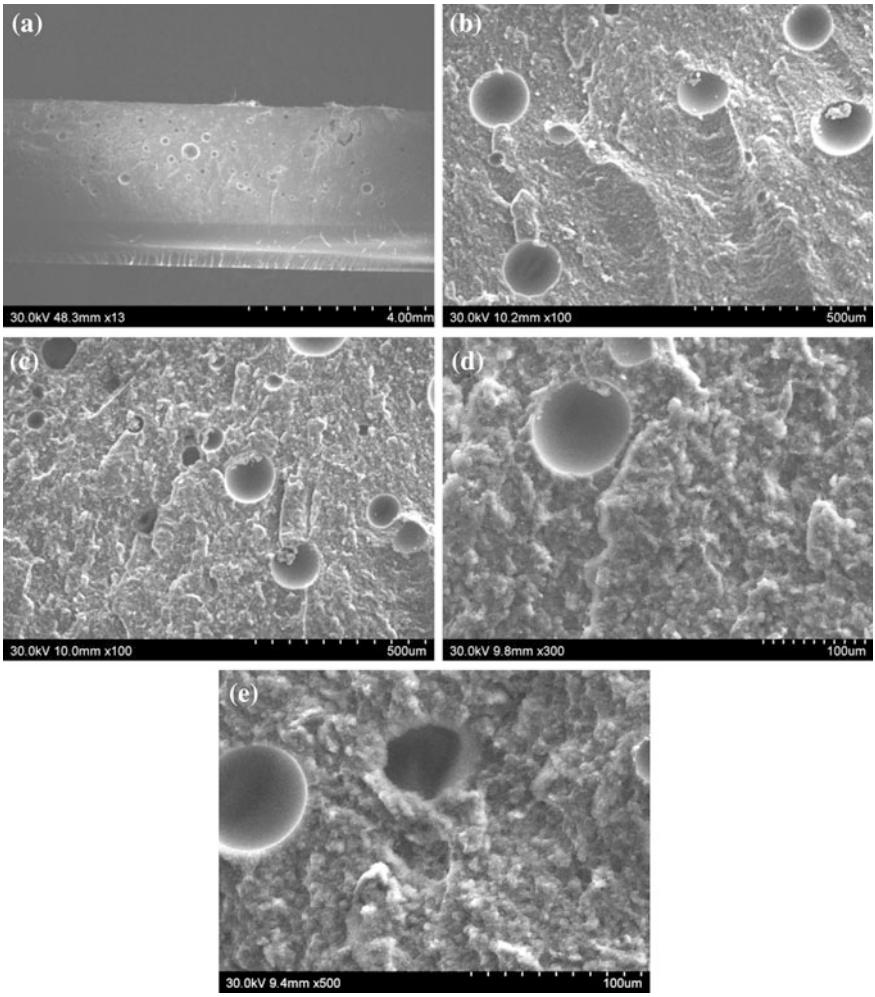

Fig. 14 SEM image of flexural tested 6.67 % NC reinforced vinyl ester composites **a** overall view, **b** location 1, **c** location 2, **c** location 3, **d** location 4, **e** location 5

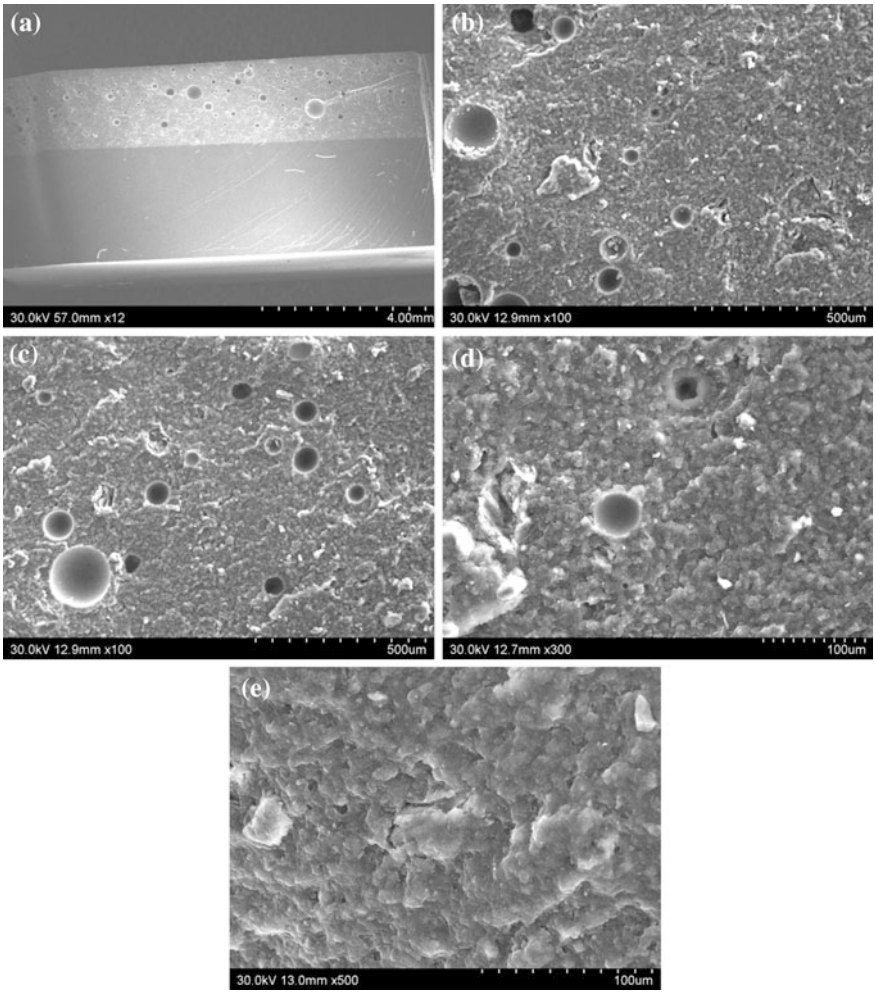

Fig. 15 SEM image of impact tested 5 % CF-6.67 % NC reinforced vinyl ester composites **a** overall view, **b** location 1, **c** location 2, **c** location 3, **d** location 4, **e** location 5

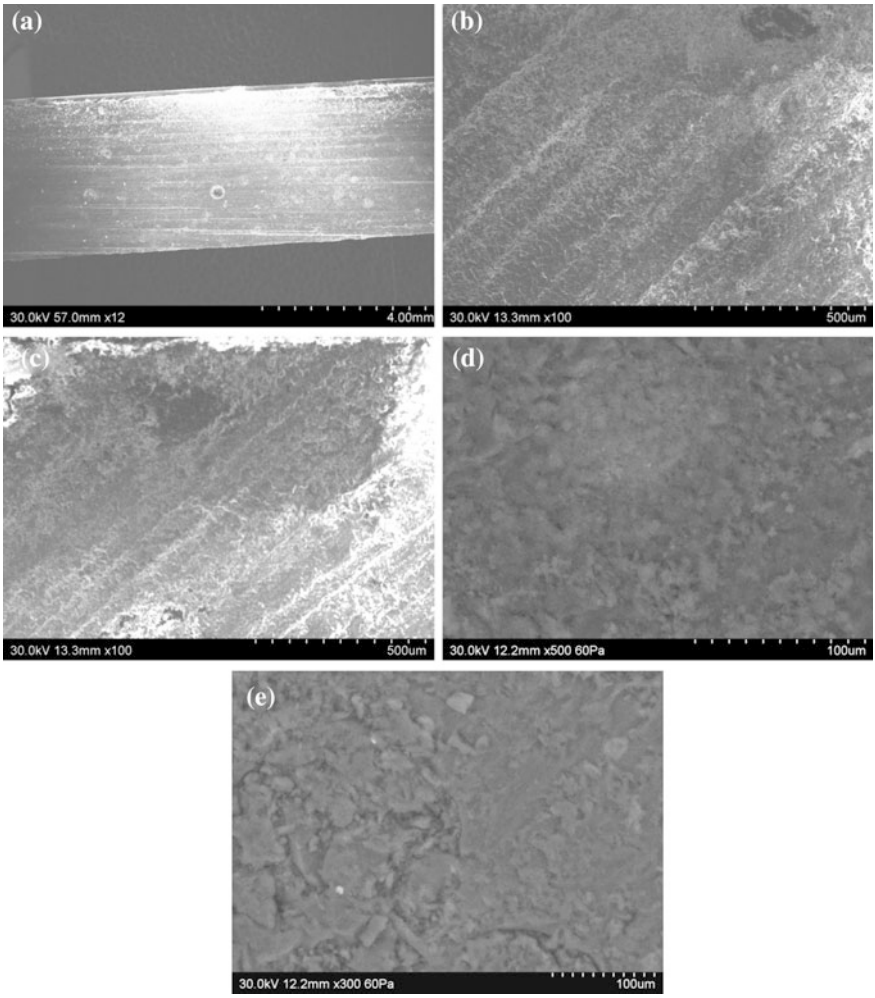

Fig. 16 SEM image of impact tested 6.67 % NC reinforced vinyl ester composites **a** overall view, **b** location 1, **c** location 2, **c** location 3, **d** location 4, **e** location 5

4 Conclusions

CF-NC, NC particulate reinforced vinyl ester composites have successfully prepared by two stage hand/wet layup technique. This process of manufacturing has ensured complete presence of reinforcement in the matrix. Out of all the composites experimentally studied in this work, composites with CF-NC have exhibited superior performance when compared with NC alone reinforced composites. Mechanical

stirring of the resin/reinforcement(s) mixture result in better distribution/mixing of reinforcements in the matrix.

Though the present attempt and investigation has given some satisfactory results, there is a serious emergent need to thoroughly investigate the MTSHLM process to enhance mechanical performance of the composites along with uniform distribution of reinforcements.

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