Article

Multi-Scale Study on Mechanical Property and Strength of New Green Sand (Poly Lactic Acid) as Replacement of Fine Aggregate in Concrete Mix

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Abstract: Polylactic acid (PLA) has made inroads in the commercial market segment with many unique characteristics. To list a few, such as tenacity, low flame rate, moisture regain percentage, loss of ignition percentage, combustion heat, UV resistance, elastic recovery, and higher melting point, make PLA a predominant material in the commercial market. This study is an attempt to test the feasibility of PLA's mechanical property and strength aspects with cement mix. An article published on biodegradability aspects backed up by the essential preliminary strength and physical test results is discussed in detail in this manuscript. The work focuses on the multi-scale study along with mechanical properties and strengths to evaluate the elemental characteristics. Thermo gravimetric analysis revealed that PLA would hold inclusion into construction applications either in granular form or filament. Differential Scanning Calorimetry (DSC) found that PLA in filament form is the best inclusion material for construction applications. However, fiber's tenacity has to be checked, as currently available filaments in the market do not have high tenacity value. From EDX(Energy-dispersive X-ray Spectroscopy) reports, 30% inclusion of PLA as a replacement for fine aggregate has constituent members as Calcium carbonate(CaCO_3), Silica(SiO_2), and Wollastonite (CaK) resulted in the best composition among the rest. FESEM images revealed that proper gradation in size, PLA granular form's rough surface, or filament form would enhance the mechanical/physical behavior or even PLA's chemical behavior.

Keywords: poly lactic acid(PLA); TGA; FTIR; DSC; FESEM; EDX

1. Introduction

As per plastic insight [1], by 2020, the global polylactic acid (PLA) market will reach a milestone of more than US\$ 5 billion. The usage has currently catered to many diversified domains such as medical, food packaging, textile, agricultural, consumer electronics, personal care, etc. As in India, the usage is limited to 1% of global utilization in 2015 [2]. In India, solid waste generation has opened a Pandora of opportunity boxes to recycle the dry solid waste with the best outputs.

Today, material characterization has become part of the research for identifying the critical parameters such as micro/nanostructural study with SEM (Scanning Electron Microscopy)/FESEM (Field Emission Scanning Electron Microscopy)/TEM (Transmission Electron Microscopy) help. Loss of volatile components viz. moisture, solvent, and monomers, decomposition, noticing the residue composition using Thermogravimetric analysis (TGA), Enthalpy, melting point and specific heat capacity by Differential Scanning Calorimetry (DSC), to arrive at an infrared spectrum of absorption or emission of all three phases such as a solid, liquid, or gaseous material using Fourier Transform Infrared Spectroscopy (FTIR). Current generation TGA DSC has a wide range of measurement techniques that performed dynamically using a linear temperature ramp or isothermally [3]. There were quite a good number of studies focused on micro characterizations. To name a few such as coal bottom ash, crushed rock sand, copper slag, foundry sand, recycled fine aggregates, and sand-based cemented paste backfill material (SCPB) [4]. X-ray diffraction for coal bottom ash (CSH) results in poor legibility of diffraction peak due to CSH's lower crystallinity [5]. Granite powder inclusion in 30% to 40% of mortar resulted in the development of Calcium Aluminate Silicate Hydrates (C-A-S-H), and its band increased to more than 1000/cm [6]. Marble waste in mortar after replaced with dolomite depicted higher peaks of calcite phases [7]. However, SEM images have shown a cluster of calcium and hydrates for copper slag inclusion in a mortar [8]. In marble powder, more porosity, crack structures, and fractures were observed [9]. For bottom ash, permeable vacant spots raise from 21.9% to 37.05% for introducing 100% inclusion of ash concerning to control mix [10]. SEM images illustrate needle-like structures and C-S-H gel distributed uniformly in the concrete mix [11]. The fine recycled aggregate showed low durability due to chemical attack and was more porous than the control mix [12]. Thermal conductivity depleted gradually to 68% for coal bottom ash with 100% inclusion [13]; for [10] it recorded 68.61% due to an increase in the number of pores due to CBA (Coal Bottom Ash) inclusion, they provide heat dissipation option to surrounding regions [14]. These conditions are made in-roads for interior coating applications to develop a "Green Building" using waste material [13].

Objectives of the Proposed Research

The proposed work aims to quantify PLA as a substitute material to fine aggregates in cement mortars in proportions ranging between 0 to 50 percentages through an exhaustive technical evaluation as per the ASTM and BIS standard test procedures. The presented work's immediate fallout intends to develop sustainable alternatives to river sand as a construction material. Later, to focus on the material characterization study to cover various aspects such as SEM, FTIR, DSC, TGA, and XRD (X-Ray Diffraction).

2. Materials and Method

2.1. Material-PLA

The granular structure of PLA, as shown in Figure 1 along with 3D printing filament, with its physical properties, is presented in Table 1, while associated mechanical properties are depicted in Table 2. The cement used for the present study confirmed Indian standards (ACC High-Performance Cement), while local fine and coarse aggregates were considered raw ingredients for concrete preparation. A physical test on cleaned fine and coarse aggregates was conducted as per IS383-2016. Water used in the concrete was confirming to IS 456: 2000.

Figure 1. Poly(lactic acid) (PLA) granules and Filament.

Table 1. Physical properties of PLA.

Sl. No	Properties	PLA
1	Specific gravity	1.25
2	Water absorption	Nil
3	Bulk density	1250 kg/m ³
4	Colour	White
5	Particle size	1.18 mm–4.75 mm
6	Shape	Rounded
7	Texture	Smooth

Table 2. Mechanical Properties of PLA.

Properties	Value
Density	1.25 kg/m ³
Melt flow rate	2.4–4.3 g/10 min
Haze	2.2
Yellowness index	20–60
Heat deflection temperature	40–45 °C
VICAT softening point	56 °C
Glass transition temperature, T _g	55–56 °C
Melting point, T _m	120–170 °C
Continuous service temperature	–20 to 40 °C
Tensile strength	55 (±10%) MPa
Yield tensile strength	53 MPa
Ultimate tensile strength	73 MPa
Tensile Modulus	3150 MPa
Flexural strength	55 MPa
Flexural modulus	350–450 MPa
Yield strength	106 MPa
Young's modulus	70 MPa
Shear modulus	1975 (±7%) MPa
Elastic modulus	1287 MPa
Elongation at yield	10–100
Elongation at break	9.3 (±7) %
Poisson's ratio	0.36
Percent of elongation	11.3%
Unnotched Izod Impact	195 J/m
Notch Izod Impact	26 J/m
Rockwell hardness	88 J/m

Superplasticizer

The superplasticizer used for the present study was conforming to IS 9103: 1999 and Conplast 430 superplasticizer as the water reducer. FOSROC Chemicals India Pvt Ltd., Bengaluru, is the

manufacturing company. The reported specific gravity of it was 1.220 to 1.225 at 30 °C. Its chloride content was negligible and observed as brown.

2.2. Methodology

The process map of presented work highlighted in Figure 2 briefs on entire career accompanied in the adopted research methodology.

Figure 2. Process mapping of entire work with Scheme of Methodology.

The Indian agricultural sector's immense potential charted the country as the second-largest producer of sugar cane, potato, and corn during 2013 (word bank 2014, FAOSTAT 2013) with year-on-year production levels exhibiting the rising trend the current year. The massive cultivation of these crops forms the resource bank of raw materials to explore PLA's large scale production. The waste generated from crop harvesting to the final usage will result in PLA as a by-product to substitute for the ecological deterrent river sand.

2.3. Work Carried Out

2.3.1. Preliminary Work

Sieving of aggregates carried out to get the desired gradation and impurities separated. Mix design of M20 and M30 considered for the preparation and testing of coupons. The relevant details for the trial mixing approach to get the expected proportion of ingredients and workability for concrete are discussed in Table 3.

Table 3. Details of ingredients along with compositions.

	Materials	Replacement of Fine Aggregate by PLA (%)			
		0%	10%	30%	50%
M20 (1:1.65:2.8) $\frac{W}{C} = 0.5$	Cement (Kg/m ³)	350	350	350	350
	River Sand (Kg/m ³)	577.5	519.65	404.17	288.69
	PLA granules (Kg/m ³)	0	30.325	90.975	151.625
	Coarse Aggregate (Kg m ³)	980	980	980	980
	Admixture	0.73%	0.73%	0.73%	0.73%
	Water (mL)	175	175	175	175
	Materials	Replacement of Fine Aggregate by PLA (%)			
		0%	10%	30%	50%
M30 (1:1.62:2.65) $\frac{W}{C} = 0.38$	Cement (Kg/m ³)	420	420	420	420
	River Sand (Kg/m ³)	680	611.97	475.98	339.983
	PLA granules (Kg/m ³)	0	31.71	107.138	178.56
	Coarse Aggregate (Kg/m ³)	1113	1113	1113	1113
	Admixture	1.20%	1.20%	1.20%	1.20%
	Water (mL)	159.6	159.6	159.6	159.6

2.3.2. Preparation of Coupons

They are mixing concrete, casting of coupons, demolding, and their curing carried out as per BIS (Bureau of Indian Standards). Coupons cast as shown in Figure 3.

Figure 3. (a) Specimen compaction, (b). casted cube, (c) PLA used coupons, (d) casted coupons.

2.3.3. Tests and Specimen Sizes

The extensive work comprised 146 coupons preparation for various sets of tests. The size and shape of the present work's specimens were as recommended by the Indian standard.

The casted test samples were sized and configured into the geometries of the cube, cylinder, and beam elements that aggregated to a total of 146 coupons, as presented in Table 4 with varying proportions of PLA. The investigations utilized three sets of coupons per test case with separation of M20 and M30 mix cases as detailed out in Table 4 as per the requirements of various tests.

Table 4. Total of 146 specimens for various sets of tests.

Specimen Type	Sizes and Respective Tests	Total No. of Specimens
Cubes	10 cm × 10 cm × 10 cm (Compressive strength, Water absorption, and Density)	48
	15 cm × 15 cm × 15 cm (Water Permeability)	8
	7.06 cm × 7.06 cm × 7.06 cm (Compressive strength, Water absorption, and Density)	24
Cylinders	15 cm dia, 30 cm height (Split Tensile)	32
	15 cm dia, 6 cm Height (Impact)	16
Beams	10 cm × 10 cm × 50 cm (Flexural strength)	18

2.3.4. Sieve Analysis

(a) Fine aggregate:

Sand used for the present study confirmed to Zone 3, and its fineness modulus is 3.16. It was coarse sand and light brown. Sieve analysis and physical properties of fine aggregate tested as per IS383-2016.

(b) Coarse aggregate

The coarse aggregate used for the present study was light grey. Sieve analysis details were as depicted in Table 5, and the physical properties of coarse aggregate under IS 383-2016.

Table 5. PLA sieve analysis (weight of sample taken: 200 g).

ISSieve mm	Weight Retained in Grams	Percentage of Weight Owned in Grams	Cumulative Percentage Retained	Percentage of Passing
10 mm	0	0	0	100
4.75 mm	0	0	0	100
2.36 mm	193	96.5	96.5	3.5
1.18 mm	7	3.5	100	0
600 Micron	0	0		
300 Micron	0	0		
150 Micron	0	0		
75 Micron	0	0		
Pan	0	0		

(c) PLA granules

PLA granules replaced the fine aggregates; as the granules fabricated had similar size, they filtered through a 4.75 mm sieve. The colours of the granules were white. Sieve analysis and physical properties of PLA granules tested for IS 383-2016 and results were obtained, as shown below in Table 5. PLA granules were laying between 4.75 mm to 1.18 mm. PLA was round in shape with smooth texture and when it comes to grading, it fails in all the zones.

3. Results and Discussion

The specimens were being tested for fresh and hardened properties. The following were the tests carried out with various coupons as per ASTM and BIS.

3.1. Slump Test

The slump test was being conducted according to IS: 1199-1959, and the results were mentioned in below Table 6.

Table 6. Slump Test results.

Replacement of Fine Aggregate by PLA	Slump(mm)	
	M20	M30
0%	70	50
10%	80	65
30%	95	85
50%	Collapse	Collapse

With the increase in the size of aggregates, workability enhances. The larger the extent of aggregates, the lesser will be the surface area available; hence, a less amount of water is required for wetting the surface of aggregates—a less amount of paste required for the lubrication of the surface to reduce the internal friction. Since PLA granules were coarser than fine aggregate and having lesser water absorption property than the river sand, water and paste will remain in excess in lower replacement percentages, leading to the formation of a fatty and cohesive mix, which provides better workability.

PLA granules were coarser and poorly graded. Thus, the formation of paste using finer particles of fine aggregate decreases at higher replacement percentages. It results in segregation in the concrete and sliding of aggregate particles. As a result of this, the slump got collapsed. Figure 4 depicts the slump test M20 and M30 grade of specimens. The concrete mix paste formation was also less at higher replacement percentages. It causes an excess of water in the mix, resulting in segregation.

Figure 4. Slump test for M20 and M30 grade.

3.2. Compressive Strength

The specimens were tested for compressive strength under IS 516-1959 and the results are shown below in Table 7. The compressive strength at 10% replacement gave 46.84 MPa (7 days) and 49.42 MPa (28 days) for M30 grade concrete compared with sawdust-25 MPa and 36 MPa (Ahmed et al., 2018 [15]). Bagasse fiber-48 MPa and 51 MPa (Moretti et al., 2018 [16]), used foundry sand-18 MPa and 25 MPa (Manoharan et al., 2018 [17]), treated oil sand waste 2 MPa and 2.8 MPa (Mneina et al., 2018 [18]), palm oil clinker powder -30 MPa, and 30 MPa (Nayaka et al., 2018 [19]), respectively.

Table 7. M20 and M30 grade results with 7 days and 28 days curing.

Days of Curing	Replacement of Fine Aggregate by PLA	M20		M30	
		Compressive Strength (MPa)	Variation in Strength	Compressive Strength (MPa)	Variation in Strength
7 Days	0%	19.37	0%	28.40	0%
	10%	38.01	96.19%	46.84	65.00%
	30%	24.03	24.03%	32.87	15.74%
	50%	14.96	-22.78%	24.53	-13.63%
28 Days	0%	30.90	0%	38.99	0%
	10%	44.27	43.25%	49.42	26.75%
	30%	30.78	0.39%	38.38	-1.56%
	50%	19.37	-37.30%	26.85	-31.14%

From the above data M20 and M30 concrete, maximum compressive strength was achieved by replacing 10% of fine aggregate with PLA granules. By replacing 30% of fine aggregate with PLA granules, strength matches that of control concrete. Voids formation in 10% usage of PLA in concrete didn't cause much impact on the strength. As PLA granules were in small quantities, they scattered throughout the volume of concrete.

Lack of bonding by the PLA granules with other concrete particles resulted in PLA having a smooth glassy texture. Larger size and round shape of PLA results in a decrease of surface area for bonding and an increase in void formation. A less amount of water is required for wetting the surface of aggregates. However, PLA granules are the synthesized hydrophobic material that will absorb very negligible water particles compared to river sand. On the other side, the coupons were kept for four weeks in water, resulting in PLA's outer surface going into degradation, yielding lower strength. An increased water/cement ratio and segregation are the few more parameters that caused the strength depletion.

PLA granules were coarser and poorly graded. Higher replacement percentages of PLA results in a lack of paste formation in the mix and segregation. A percentage increase in 7 days strength by the inclusion of PLA in concrete is more than that of 28 days, as depicted in Figure 5. This is due to the PLA granule's outer layer degradation.

Figure 5. Compressive strength test for M20 and M30 grade for 7/28 days curing.

3.3. Split Tensile Strength

The specimens were being tested for split tensile strength under IS 516-1959 and the results are discussed in Table 8.

Table 8. M20 grade results with 7 days and 28 days curing.

Days of Curing	Replacement of Fine Aggregate by PLA	M20		M30	
		Split Tensile Strength (MPa)	Variation in Strength	Split Tensile Strength (MPa)	Variation in Strength (%)
7 Days	0%	1.457	0	2.115	0
	10%	1.664	14.20	2.635	24.59
	30%	1.318	−9.54	2.011	−4.92
	50%	1.04	−28.62	1.56	−26.24
28 Days	0%	2.653	0	3.606	0
	10%	2.913	9.80	3.92	8.70
	30%	2.254	−15.04	3.433	−4.80
	50%	1.231	−53.60	2.323	−35.58

The split tensile strength at 10% replacement yielded 2.635 MPa (7 days) and 3.92 MPa (28 days) for M30 grade concrete, which in comparison to used foundry sand −1.9 MPa and 2.3 MPa (Manoharan et al., 2018), palm oil clinker powder −2.4 MPa and 3.3 MPa (Nayaka et al., 2018), respectively.

Figure 6, M20 and M30 concrete coupon test shows that maximum split tensile strength is achieved by replacing 10% of fine aggregate with PLA granules. Split tensile strength decreases with the increase in percentage replacement above 10%. Since PLA has higher tensile strength, its inclusion in concrete around 10% of fine aggregate weight imparts good tensile strength.

Figure 6. Split tensile strength test results for M20 and M30.

3.4. Flexural Strength

The coupon tests were conducted for flexural strength under IS 516-1959, and the results were mentioned in Table 9.

Table 9. M20 grade results with 7 days and 28 days curing.

Grade of Concrete	Replacement of Fine Aggregate by PLA	Average Flexural Strength (MPa)	Variation in Strength (%)
M20	0%	3.48	0
	10%	4.36	25.29
	30%	3.92	12.64
	50%	2.96	−14.94
M30	0%	4.46	0
	10%	5.74	28.70
	30%	4.38	−1.79
	50%	3.08	−30.94

(a) Sample with M20 grade (Load vs. Deflection)

Table 9 depicts the tabulated data about deformation versus load applied to reveal that 10% inclusion of Fine Aggregate by PLA yielded an average load of 10.9 kN. Figure 7 illustrates similar notions in the form of a graph.

The flexural strength at 10% replacement for M30 grade reported a load bearing of 14.4 kN, which is by far the best in comparison to used foundry sand −5.05 kN (Manoharan et al., 2018), palm oil clinker powder −4.5 kN (Nayaka et al., 2018), respectively.

Figure 7. Flexural strength test M20 grade for all cases.

(b) Sample with M30 grade (Load vs. Deflection)

Figure 8 revealed that the M30 mix resisted 5.74 MPa against the M20 mix that took a load of 4.4 MPa owing to PLA outer granule degradation at the surface during a 28-day cure duration. The microstructural FESEM provides further insights into the strength gain mechanism concerning PLA's proportion in concrete.

Figure 8. Flexural strength test M30 grade for all cases.

(c) Sample with M20 grade (Flexural strength vs. % replacement of FA by PLA-28 days)

From the above M20 and M30 concrete data, maximum flexural strength is achieved by replacing 10% of fine aggregate with PLA granules. Replacement of 30% fine aggregate by PLA granules still results in comparatively higher flexural strength than that of control concrete. Flexural strength decreases with the increase in percentage replacement above 30%. The amount of deflection in both M20 and M30 concrete decreases with the addition of PLA granules, but its addition increases the load-bearing capacity as shown in Figure 9.

Figure 9. Flexural strength test with percentage replacement of fine aggregate by PLA.

3.5. Water Absorption

The coupons were being tested for water absorption under BIS and the results are shown in Table 10. Water absorption percentage of M20 concrete was more significant than M30 concrete due to the following reasons.

Table 10. M20 grade results for the water absorption test.

Replacement of Fine Aggregate by PLA	Average Water Absorption (%)	
	M20	M30
0%	5.49	3.49
10%	1.97	1.12
30%	4.52	3.57
50%	5.63	4.47

Cement content in M30 concrete was more than M20 concrete, as shown in Table 9. With the increase in cement content, there were more hydration ingredients and the zone of voids increases the result in strength deficit. Thus, the capillary cavities and its diameter decreases, which results in the reduction of water absorption percentage.

The water/cement ratio in M20 concrete was more than M30 concrete. With the increase in the water content, voids are filled with water, increasing water absorption percentage. From Figure 10, at 10% replacement of fine aggregate by PLA granules, water absorption percentage reduced as

PLA (hydrophobic material) has a negligible water absorption percentage. At 10% replacement of fine aggregate by PLA granules, PLA granules were in less quantity. Hence, the voids were less, which reduces the water absorption percentage. With the increase in the replacement percentage, the aggregates' water absorption decreased, but the number of voids increased, which triggered a higher water absorption percentage.

Figure 10. Water absorption test.

3.6. Density

The coupons were being tested for density under BIS, and the results are shown in Table 11.

Table 11. M20 and M30 grade results for density test.

Replacement of Fine Aggregate by PLA	Average Dry Density (Kg/m ³)	
	M20	M30
0%	2425.0	2582.5
10%	2537.5	2672.5
30%	2387.5	2455.0
50%	2352.5	2405.0

The density of concrete of any proportion is:

$$\text{Density} = (\text{Mass of concrete specimen} / \text{Volume of specimen}) \text{ in kg/m}^3$$

Cement content in M30 concrete was more than M20 concrete, as illustrated in Table 9. With the increase in cement content, there were more hydration products and the filling up of voids increases. Thus, the capillary cavities and its diameter decreases, which was further removed by proper compaction. Hence, density value shoots up. The water/cement ratio in M20 concrete was more than M30 concrete. Figure 11 shows that with the increase in the water content, voids are filled with water. This water will not escape on compaction, which causes a decrease in the dry density.

Figure 11. Density test.

M30 concrete density was more significant than M20 concrete due to the following reasons. PLA granules were lighter than existing, using alternative sand materials. With the increase in the percentage replacement, the concrete will become lighter and decrease dry density. As the replacement percentage increases, there will be more voids, and paste formation decreases. Thus, the voids remain unfilled, which reduces the dry density. The deadweight of concrete reduces as the % of PLA increases. This causes the reduction in deadweight of structure, the dimensions of beams, columns, etc. compared to standard concrete. Hence, it comes out to be an economically viable solution. In the present study, density was maximum for 10% usage of PLA granules. In this composition, voids were significantly lesser than other higher percentage usage of PLA and it is almost equal to that of control concrete. But the increase in density for 10% usage may be due to good compaction.

3.7. Water Permeability

The coupons for water permeability under IS 3085-1965, and the results are tabulated in Table 12.

The coefficient of permeability of M_G20 concrete was more than M_G30 concrete, as mentioned in Table 12. This was due to an increase in the cement content and lesser water/cement ratio in M_G30 concrete. M_G20 concrete had a higher coefficient of permeability because of the numbers, and the diameter of capillary cavities in the concrete increase with the water/cement ratio increases. For a 10% replacement of fine aggregate by PLA, the coefficient of water permeability reaches its lowest value. With the rise in percentage replacement of fine aggregate by PLA above 10%, the coefficient of permeability also increases, due to the formation of a large number of voids and lack of matrix in the concrete as discussed in Figures 12 and 13.

Table 12. M20 and M30 grade water permeability test.

Grade of Concrete	Replacement of Fine Aggregate by PLA	Water Collected (mL)	Time Elapsed	Coefficient of Permeability (cm/s)	Average Coefficient of Permeability (cm/s)
M20	0%	2306	100 h	8.54×10^{-5}	8.67×10^{-5}
		2372		8.79×10^{-5}	
	10%	2228		8.25×10^{-5}	8.29×10^{-5}
		2250		8.33×10^{-5}	
	30%	2494		9.24×10^{-5}	9.54×10^{-5}
		2588		9.59×10^{-5}	
	50%	2734		10.13×10^{-5}	10.37×10^{-5}
		2866		10.62×10^{-5}	
	0%	1782		6.60×10^{-5}	6.53×10^{-5}
		1744		6.46×10^{-5}	
M30	10%	1702		6.30×10^{-5}	6.28×10^{-5}
		1688		6.25×10^{-5}	
	30%	1862		6.90×10^{-5}	6.80×10^{-5}
		1808		6.70×10^{-5}	
	50%	1990		7.37×10^{-5}	7.21×10^{-5}
		1902		7.04×10^{-5}

Figure 12. (a) and (b) show sealing the specimen inside the permeability cell.

Figure 13. Results on water permeability test.

3.8. Impact Resistance Test

The impact test carried using a drop weight hammer for cylindrical disc using a drop weight testing machine. The cylindrical discs of 150 mm diameter and 60 mm thick were placed on the impact testing machine's base plate and then struck with repeated blows. The former value measures the number of blows required to initiate a visible crack, as shown in Figure 14.

Figure 14. (a) Impact test machine. (b) Coupon before test. (c) Failure of coupon.

However, the latter measures the number of blows required to initiate and propagate cracks until ultimate failure. The number of blows needed to cause the specimen's complete failure was observed as the impact failure strength in Table 13. Impact resistance of the sample was determined at 28 days curing period and the required standard specimen molds built by using PVC pipes and GI (galvanized iron) sheets.

Table 13. M20 and M30 grade Impact resistance test.

Grade of Concrete	Replacement of Fine Aggregate by PLA	Average First Crack Strength (Blows)	Average Failure Strength (Blows)
M20	0%	85	89
	10%	87	92
	30%	95	98
	50%	20	23
	0%	99	102
M30	10%	109	112
	30%	124	126
	50%	38	40

Figure 15 infers that the impact resistance of concrete slightly increases with the increase in percentage replacement of fine aggregate by PLA up to 30%. Beyond 30% replacement, impact resistance decreases significantly due to the lesser bonding strength between the particles in concrete. The impact resistance of M_G20 concrete was more than M_G30 concrete. This was due to an increase in the cement content and lesser W/C ratio in M_G30 concrete.

Figure 15. Impact resistance results for M20 grade.

3.9. Wrapping the Concrete Beam with PLA Sheet

From the previous studies on PLA, it is clear that PLA has higher flexural strength. When PLA granules remain embedded in concrete by replacing fine aggregate in the present study, it also showed increased flexural strength up to 30% replacement level. This property of PLA is effectively utilized by wrapping the concrete beam with PLA in a sheet.

In the present study, wrapping strap at the mid-span, 200 mm length plate placed at 100 mm on either side from the center of the beam, as shown in Figure 16. The size of the beam used was 100 mm × 100 mm × 500 mm, and the size of the PLA sheet was 200 mm in length, 100 mm width, and 2 mm thick.

Figure 16. The bottom face of the wrapping beam.

A 3D printing machine prepared PLA sheet at KLE Technological University. The beam used for wrapping was an M20 grade concrete beam, and for the adhesion of PLA sheet to concrete beam, an adhesive Epoxy resin is used. After the wrapping of the PLA sheet to a concrete beam, it was allowed to cure for one day and then loaded on a Universal testing machine to determine the beam's flexural strength, as shown in Figure 17.

Figure 17. PLA plate wrapped concrete beam before testing.

The adhesive agent developed by mixing Epoxy resin with a hardener in a ratio of 9:1.

Name of the resin: -Lapox L-12

Name of the hardener: -Lapox K-6

Mixing proportion: -[L-12:K-6:9:1] Curing: 14 h–24 h at 25 °C

From the above data, it is clear that load carrying capacity and ultimate deflection of the beam increases by wrapping the concrete beam with PLA plate. Line of fracture occurs at the periphery of the PLA plate as shown in Figure 17. This shows that the PLA plate resists the shaft's failure in the maximum bending moment region and fails in the unwrapped area.

From Table 14, flexural strength is comparatively higher for the case of wrapped than later. Line of fracture occurs at a distance of 10 cm from the nearest support. The obtained result was rejected due to the guidelines of IS 516:1959. It was observed that the PLA plate used for wrapping was not subjected to any damage or deformation. The experiment has proved that much scope exists to wrap the beam with PLA plate throughout the tension surface to get comparatively better results. Figure 18 depicts the importance of covering the PLA plate at the mid-span, with more than a 30% increase in its load-bearing capacity.

Table 14. Flexural strength results of wrapped and unwrapped beams.

Deflection(mm)	Load in kN	
	Without Wrapping	With Wrapping
0	0	0
0.1	1.3	1.7
0.2	1.8	1.8
0.3	2.1	2.1
0.4	2.6	3.2
0.5	3.1	3.7
0.6	3.2	4.3
0.7	3.9	5.2
0.8	4.6	5.3
0.9	5	5.5
1	5.1	5.6
1.1	5.4	5.9
1.2	5.8	7.1
1.3	6.7	7.8
1.4	6.8	8.5
1.5	7.5	9.5
1.6	8.2	10.6
1.7	9	10.9
1.8	-	11.1
		11.3

Figure 18. vs. Deflection curve of wrapped and unwrapped beams.

4. Characterization Study

A micro characterization study has become an integral part of the research study, as without these results, it can't be confronted with any justification. There are quite a good number of experimental studies that are available today to look for advanced outcomes for proper reasons. A few of such tasks were undertaken, discussed here as follows.

4.1. Thermogravimetric Analysis (TGA) for PLA Granular Form (Pristine)

Thermogravimetric analysis (TGA) measures the loss of a sample mass when subjected to heat, cold, or constant temperature. It represents the mass loss curve, in which abscissa illustrates temperature in degree Celsius and ordinate with weight percentage. The weight loss of coupons is subjected to TGA via five major steps. In the first step, loss of volatile components such as moisture, monomers, and solvents. The second step focuses on the specimen; in the third step, the atmosphere switched from nitrogen to oxygen. The fourth step is to combust carbon in endothermic or exothermic conditions—finally, an inert inorganic residue of ash fillers. Aluminum crucibles are needed to hold the specimens and working temperature of up to 600 °C. The characterization was applied to PLA in granular form, PLA in filament form, and PLA in wafer form derived from degradation of the matrix in concrete mortar.

From Figure 19, the TGA curve segregated into four steps. In first, there was up to 41.83 °C loss of volatile components such as moisture solvent and monomers. Decomposition of PLA carbon and its allied members at 59.05 °C occurred in step 2. In the third step, the atmosphere switched from nitrogen to oxygen to cater to carbon's combustion in the next step. It finally resulted in the inert inorganic residue of ash fillers or other impurities. Evaluation of a pyrolysis weight loss step yields a polymer content of 98.8%; thus, 1.2% weight loss for the granular form is recorded.

Figure 19. Weight loss vs. temperature curve for PLA granules.

4.1.1. PLA in the Filament (Wired) Form

PLA, in its filament (wired) setup, was extruded to use for 3D printing applications. When the same is subjected to TGA, it resulted in comparatively less amount of carbon black percentage (approximately 1%). Figure 20 shows a minimal amount of impurity. Temperature ramps were used to measure temperature-dependent processes such as loss of moisture, composition, and chemical reaction. PLA in wired form weighing about 5.013 mg was heated at a heating rate of 10 °C/min from 20 °C to 200 °C. The glass transition point T_g and melting point T_m was measured.

Figure 20. Weight loss vs. temperature curve for wired PLA.

4.1.2. Reacted Layer on PLA Granules

As depicted in Figure 21, PLA in its diaphragm or wafer form does subject to a chemical reaction with concrete mortar for 28 days of curing. When the coupon is at seven days time limit, there was no sign of degradation in its outer peels of PLA granule, but a full cured sample yielded a white layer in the form of a wafer. The wafer, as shown in Figure 22, reveals that the entire process of moisture desorption and decomposition is carried out in two steps. The percentage of mass loss observed was 25%, yielding as expected 75% before it flattens with respect to abscissa measure with the temperature at 200 °C. Among the three cases, it is interesting to know what parameters caused the weight loss of PLA and any chemical reaction with cement mortar or water soaking (submerging in water) to convert hydrophilic to hydrophobic areas of interest. FT-IR and DSC tests will reveal facts and figures about the work.

Reacted PLA layer degrades in three stages. The first stage (23.52 °C–40.89 °C) is the loss of surface water and di-hydroxylation of the material with a weight loss of 0.93% in this period. The second transition (40.89 °C–100.82 °C) is related to the degradation of cellulosic substances such as hemicelluloses and cellulose with a weight loss of 21.06%. The third stage (100.82–120 °C) of the decomposition is due to the degradation of non-cellulosic materials with a weight loss of 0.863%. From 120 °C onwards, the relationship between weight and temperature is almost linear.

Figure 21. PLA outer layer (wafer or diaphragm) during the curing period.

Figure 22. Weight loss vs. temperature curve for reacted PLA layer.

4.2. Differential Scanning Calorimetry (DSC)

DSC is a thermoanalytical method. Generally, polymers behave significantly when the temperature drops below T_g (more brittle) or goes up to higher temperatures than T_g (more rubbery). Hence, to select a material for a specific application, it is essential to know a composite's behavior under applied heat flow.

DSC measurements were conducted using a calibrated instrument available with a cooling attachment in inert nitrogen atmospheric conditions. Each sample was cut into tiny pieces (about 4–10 mg) and sealed in hermetic pans and lids. To have reasonable reliability of the results, three samples from different regions were evaluated by DSC for each material. The data were collected by repeated heating-cooling-heating cycles. The heating and cooling rate was 10 °C/min.

4.2.1. PLA Granules

In this analysis, the weight of the sample taken was about 7.725 mg, and the samples were heated up to 190 °C and held in a molten state for 5 min, followed by cooling down to 20 °C as shown in Figure 23.

Figure 23. Heat flow vs. temperature behavior of PLA granules.

Glass transition temperature (T_g) = 54.04 °C

Crystallization temperature (T_c) = 143.87 °C

Melting temperature (T_m) = 154.15 °C

4.2.2. PLA in Wired Form

Figure 24 shows PLA's wired form was subjected to testing through TGA, resulting in a new pictorial representation of heat flow compared to granular form. The reason might be due to the decomposition of chemical bond termination [4].

In this analysis, the weight of the sample taken was about 5.013 mg, and the samples were heated up to 190 °C and held in a molten state for 5 min, followed by cooling down to 20 °C.

Glass transition temperature (T_g) = 76.03 °C

Crystallization temperature (T_c) = 165.09 °C

Melting temperature (T_m) = 177.94 °C

Figure 24. Heat flow vs. temperature behavior of wired PLA.

4.2.3. Reacted Layer on PLA Granules

From Figure 8, it can be inferred that the concentration of PLA as it gets linked with cement causes a hydrolysis process which delivers an early degradation of coupons. The higher the percentage of inclusion of PLA combination yields less defensive and is characterized by decreased degradation temperature. This decrease in temperature results due to low bond strength and early decay of material [5].

In this analysis, the weight of the sample taken was about 4.999 mg, and the samples were heated up to 190 °C and held in a molten state for 5 min, followed by cooling down to 20 °C as shown in Figure 25 [6].

Figure 25. Heat flow vs. temperature behavior of reacted PLA layer.

Glass transition temperature (T_g) = 48.11 °C

Melting temperature (T_m) = 95.57 °C

4.3. Fourier Transform Infrared Spectroscopy (FT-IR)

4.3.1. PLA Granules

The FTIR spectroscopy deals with the study of functional clusters such as PLA granules at various weight percentage inclusions to diagnose the effect of PLA with concrete and any chemical changes happening in the surrounding region of bonding. Figure 26 infers that a broad peak of 3437 cm^{-1} was observed, indicating a hydroxyl group with a pristine sample subjected to testing with lesser lignin and hemicellulose content than wired filament form. The effect is shown when compared with Kenaf fiber with treated 3324 cm^{-1} and untreated 3318 cm^{-1} [7]. Further, the characteristics were compared with PLA, PLA/Kenaf (Untreated), PLA/Kenaf (Treated), and PLA/Kenaf (Treated)/EJO samples [7]. A biocomposite coupon interpreting a combination of PLA and fiber are mixing peak. Because concrete will undergo hydration and calcination, the expected result will affect a blend of PLA and concrete coupons. However, as for all three samples besides PLA spectrum, they had approximately similar wavelengths compared to PLA's except for the difference in the peak intensity in the C=O stretching and C–O stretching for PLA as shown in Table 15.

Figure 26. Bands in IR spectrum of PLA granules.

Table 15. Peak positions in IR spectrum for PLA granules.

Bonding	Type	Characteristic Peak Positions (cm ⁻¹)
-C-C-	Stretching	874.73
-OH	Bending	3437.83
-C-O-	Stretching	1090.65
-CH ₃	Asymmetric Bending	1454.09
	Symmetric Bending	1430.25
	Asymmetric Stretching	2996.68
	Symmetric Stretching	2921.69
-C=O-	Symmetric Bending	1636.30
-C=O	Carbonyl Stretching	1756.64
-CH-	Symmetric bending	2500
	Asymmetric bending	2851.79
-CH-	Symmetric bending deformation	1386.29
	Asymmetric bending deformation	1365
C=C	Stretching	1590.02

4.3.2. PLA in Wired Form

PLA wired (filament) form does depict similar peak regions as granules with slight wavelength variation. The part shown in Figure 27 with the hydroxyl group is varied with ± 3 cm⁻¹. Table 16 indicates the deviation of bonding with type and characteristic peak position study.

Figure 27. Bands in IR spectrum of PLA in wired form.

Table 16. Peak positions in IR spectrum for PLA in filament form.

Bonding	Type	Characteristic Peak Positions (cm ⁻¹)
-C-C-	Stretching	875.26
-OH	Bending	3434.34
-C-O-	Stretching	1091.59
-CH ₃	Asymmetric Bending	1453.8
	Symmetric Bending	1426.38
-C=O-	Symmetric Bending	1591.46
-C=O	Carbonyl Stretching	1754.48
-CH-	Symmetric bending	2853.68
	Asymmetric bending	2924.1
-CH-	Symmetric bending deformation	1386.1
	Asymmetric bending deformation	1360

4.3.3. Reacted Layer on PLA Granules

This particular model was subjected to a combination of PLA and concrete hydration or calculations process. The peak of the hydroxyl group got shifted as compared to its pristine sample tested for PLA. As the Figure 28 shows, symmetric bonding-C=O-shifted from 1591.46 (filament PLA) to 1483.96 (reacted PLA), and carbonyl stretching-C=O moved from 1754.48 (filament PLA) to 1585.79 (reacted PLA). This indicates miscibility and interaction taking place between PLA, cement, and fine aggregate. Table 17 depict further clarity on the type, bonding, and peak positions of characteristics.

Furthermore, the shift is quite naturally due to an interaction between the hydroxyl group of PLA, cement, and water through hydrogen bonding (H-bonding) interaction, resulting in enhanced morphological properties [7].

Figure 28. Bands in IR spectrum of a reacted layer on PLA granule.

Table 17. Peak positions in IR spectrum for PLA granules.

Bonding	Type	Characteristic Peak Positions (cm ⁻¹)
-C-C-	Stretching	781.16
-OH	Bending	3367.13
-C-O-	Stretching	1125.45
-CH ₃	Asymmetric Bending	1483.96
	Symmetric Bending	1404.31
	Asymmetric Stretching	2981.37
	Symmetric Stretching	2937.37
-C=O-	Symmetric Bending	1483.96
-C=O	Carbonyl Stretching	1585.79
-CH-	Symmetric bending	2754.86
	Asymmetric bending	2854.95
-CH-	Symmetric bending deformation	1404.31
	Asymmetric bending deformation	1371.38

4.4. Energy-Dispersive X-Ray Spectroscopy

EDS allows the measure/study of the elemental composition of the specimen. The EDX system may also control the SEM scanning system to collect essential distribution maps or entire line profiles.

In the present study, the samples used for FESEM and EDS are of M20 grade 28 days cured concrete. Conventional M20 grade concrete and on the concrete where fine aggregate was replaced by PLA granules partially. Replacement levels were 10%, 30%, and 50% volume of fine aggregate.

Conventional Concrete/Concrete without PLA Granules

Energy-dispersive spectra obtained from the quantitative elemental analysis on conventional concrete are shown below from Figures 29–33.

Figure 29. EDX (Energy-Dispersive X-Ray Spectroscopy) equipment (Courtesy of CMTI, Bangalore).

Figure 30. EDX spectrum of conventional concrete.

Figure 31. EDX spectrum of 10% PLA used a concrete sample.

Figure 32. EDX spectrum of 30% PLA used a concrete sample.

Figure 33. EDX spectrum of 50% PLA used a concrete sample.

- (a) 10% Replacement of Fine Aggregate by PLA Granules
- (b) 30% Replacement of Fine Aggregate by PLA Granules
- (c) 50% Replacement of Fine Aggregate by PLA Granules

Microscopy observations have been carried out on the fractured and flat surfaces of paste samples [6] after 28 days of curing, focusing on the bonded area between PLA granules and concrete matrix. Various compounds and elements like CaCO_3 , SiO_2 , Albite (Na), MgO , Al_2O_3 , SiO_2 , FeS_2 , KCl, Wollastonite (Ca), Ti, Mn, and Fe elemental analysis of a sample are shown in Table 4. A unique set of peaks are visible for each element's atomic structure. In Table 4, there are higher elevations for Ca. This may be due to the formation of C-S-H gels accelerated by SiO_2 particles. It also indicates the presence of Si, O peaks. The amount of CaCO_3 increases with the increase in the percentage replacement of PLA granules. The amount of SiO_2 , Albite, and MgO decreases due to a reduction in the amount of river sand in concrete on its reserve. The amount of Fe, Al_2O_3 , MAD-10 Feldspar, and FeS_2 decreases with the increase in the percentage replacement of PLA granules due to PLA's inert behavior with concrete and reduction in the amount of river sand in concrete on its replacement. The small crystals may be calcium sulfoaluminate, as indicated by the little Sulphur peak in the EDX spectrum [8].

From Table 18, it can be concluded that as the percentage of inclusion of PLA raised from 0% to 10%, calcium carbonate and silica content have risen steeply along with Wollastonite (CaK). From 10% to 30% increase in PLA resulted in a further rise of all these elements. This depicts that concrete mix of cement and coarse aggregate behaves in brittle form and PLA as virgin material behaves breakable, resulting in better mechanical strengths and control concrete mix 30% inclusion results were far more convincing than earlier.

Table 18. EDX spectrum details for 0, 10, 30, and 50% inclusion in concrete as replacement for fine aggregate.

[illegible]

4.5. Microstructural Study with Scanning Electron Microscopy (SEM)

The entire microstructural study with FESEM plus EDS was carried out at CMTI, Tumkur Road, Bangalore. The FESEM images were generated with higher spatial resolution, ultra-precision, and accuracy using Carl Zeiss, Sigma 300 VP series model. The detailed study, results, and discussion were made in future topics.

4.5.1. Control Concrete Mix with 0% PLA Inclusion

Control concrete mix samples with zero percent PLA inclusion were subjected to microstructural study and reveal that uniform agglomeration of cement, sand, fine aggregate, and coarse aggregate. Figure 34c depicts micro cracks observed at fine aggregate removed location.

It was clear from Figure 34e–h that a perfect homogeneous blend was arrived with control mix concrete, as fine aggregates were varied in size from 4.75 mm downsize until 300 μm . This resulted in air pocket covering at the region where coarse aggregate to cement paste bonding occurred. As cement always went for volume shrinkage, it maintained the adhesion even after 28 days of curing.

Figure 34. Cont.

Figure 34. (a–d) Fracture morphology of control concrete mix with 0% inclusion of PLA. (e–h) Fracture morphology of control concrete mix with 0% inclusion of PLA.

4.5.2. With 10%PLA Replacement for Fine Aggregate

Figure 35a–d FESEM images depict the enlarged drawing of PLA and concrete composites' internal structures. The grain size is calculated by the linear intercept method using the formula [9]. With Figure 35e–h it can be drawn such as, (e) shows the air pocket inside the PLA granular structure, even being hydrophobic as parent material, when mixed with cement and coarse aggregate, cast and kept for curing at 28 days it did not go on to degrade. Figure 35f,g illustrates the bead-like structure. These flakes have nonuniform lengths showing peaks and valleys in their sizes, providing additional space for the inclusion of water and air particles to penetrate the member and probably allow it for early degradation.

Figure 35. (a–d) Fracture morphology of control concrete mix with 10% inclusion of PLA. (e–h) Fracture morphology of control concrete mix with 10% inclusion of PLA.

4.5.3. With 30%PLA Replacement for Fine Aggregate

The grain size is calculated by the linear intercept method using the formula [9] for the sample $\times = 30\%$ inclusion of PLA, the length of rods will have a smaller size of 5–6 μm . As the percentage of PLA inclusion increases in volume when compared to an earlier case, there is more provision for

free space because PLA granules are round and glossy shine at the outer face resulting in improper bonding of concrete paste as shown in Figure 36a–d.

Figure 36. (a–d) Fracture morphology of control concrete mix with 30% inclusion of PLA. (e–h) Fracture morphology of control concrete mix with 30% inclusion of PLA.

Figure 36d illustrates the interfacial transition zone (ITZ) between cement paste and PLA. The ITZ can even be extracted from Back Scattered Image (BEI) from SEM machines [10]. Here, the work was focused only to identify the ITZ in the SEM images. Fracture behavior will initiate at the outer periphery of PLA, as a pristine sample PLA will not break that easily, allow it for transgranular failure pattern. The data depicts that mechanical tests such as compressive strength, flexural strength, split tension test, and impact test reveal that 10% inclusion of PLA was far more superior in strength than 30% inclusion [11]. From Figure 36e–h, it can be concluded that the debonding region got enhanced. In Figure 36e due to continuous loading, stresses generated at the intermediate part; this led to microcrack initiation at the grain boundary regions as Figure 36g depicts the intensification of cracks. When PLA inclusion percentage increased from 10 to 30, cement became the principal filling agent for void or air pocket regions. The behavior resulted in brittle fracture; through SEM images, it showed numerous weak zones. Due to debonding, fiber channels were visible with large pockets. Pull-outs were also visible in all directions [20,21].

4.5.4. With 50% PLA Replacement for Fine Aggregate

Figure 37a–d represents similar images, as mentioned in the 30% inclusion of PLA in the concrete matrix. As the percentage of PLA inclusion increased, resulting in less gradation of granular sizes, causing more open cavities, less cement, coarse aggregate mixture, less bonding with PLA granules. Finally, the outcome shows lowered mechanical strength.

Figure 37. Cont.

Figure 37. (a–d) Fracture morphology of control concrete mix with 50% inclusion of PLA. (e–h) Fracture morphology of control concrete mix with 50% inclusion of PLA.

Figure 37e–h shows many issues related to weak regions in the entire matrix resulting in early failure as a 50% yield with a higher degradation rate than its lower inclusion rates. The strengths dipped to an all-time low rate for flexural and impact compared to controlled concrete and successive cases. In compressive strength, the maximum load will be bear by PLA granules and are hard to break into pieces. The combination suites not so well for the split tension test, as it is subjected to loading, results in early debonding of PLA granules to cement paste. When the external load goes beyond the elastic limit of constituent materials in a matrix, the microcrack and turn crack deflection will be attained; slowly debonding initiates and rupture of the coupons will be observed.

The exhaustive bio composite characterization resulted in the following notable salient points.

- The amount of deflection in both M20 and M30 concrete decreases with the addition of PLA granules, but its addition increases the load-bearing capacity. At 10% replacement of fine aggregate by PLA granules, water absorption percentage was reduced as PLA (hydrophobic) material.
- With an increase in the replacement percentage above 10%, water absorption by the aggregate decreases, but the amount of voids increases, which increases the water absorption percentage.
- Since PLA granules were light in weight, with the increase in the percentage replacement, the concrete will become lighter, the amount of voids increases, results in a decrease in dry density.
- For a 10% replacement of FA by PLA, the coefficient of water permeability reaches its lowest value. This is because PLA will not allow the water to pass through it, and also voids are lesser in the concrete since PLA granules were in minimal quantity and were scattered throughout the volume. Beyond 10% replacement, water absorption by the aggregate decreases, but the amount of voids increases, which increases the coefficient of permeability.

- Impact resistance of concrete slightly increases with the increase in percentage replacement of fine aggregate by PLA up to 30%. Beyond 30% replacement, impact resistance decreases at a larger rate due to the lesser bonding strength between the particles in concrete.
- From the above results, it is clear that 10% to 30% replacement of fine aggregate by PLA granules results in good and satisfactory outcome in all the properties of fresh and hardened concrete.
- From the above results, it can be concluded that PLA granules undergo degradation with the outer surface with time. The failure pattern of 7 days tested specimen shows that the PLA granules were not damaged. But the PLA granules were damaged for 28 days tested coupons. This indicates that PLA granules were undergoing degradation concerning time and its strength declined.
- Load carrying capacity and ultimate deflection of the beam increases by wrapping the concrete beam with PLA plate.
- Thermogravimetric analysis revealed that PLA either in granular form or filament would hold suitable for inclusion into construction applications, provided degradation aspects for improvisation.
- From DSC, it can be inferred that PLA in filament form is the best inclusion material for construction application; however, the tenacity of fibres has to check, as currently available filaments in market does not have high tenacity value.
- From EDX reports, 30% inclusion of PLA as a replacement for fine aggregate has constituent members as Calcium carbonate (CaCO_3), Silica (SiO_2), and Wollastonite (CaK) resulted in best composition among the rest.
- The micro-structural study revealed that proper gradation in size, the rough surface of PLA granular form or filament form would definitely enhance the mechanical/physical or even chemical behavior of PLA. On the other hand, degradation is another critical issue with existing biomaterials. With help of the physical vapor deposition (PVD) method, a hydrophobic and biocompatible chemical can be coated on the outer periphery of filament or granular form.

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