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Development and Machining Analysis of Hydroxyapatite and Polypropylene Composite for Biomedical Applications

Dr. Amit Kumar Mehar*, Sreekanth Kotni

*Associate Professor, Department of Mechanical Engineering, Raghu Engineering College, Visakhapatnam, India
Associate Professor, Department of Mechanical Engineering, Raghu Engineering College, Visakhapatnam, India

ABSTRACT: Synthetic hydroxyapatite (HAp) particulate and thermoplastic Polypropylene (PP) polymerare produced for biomedical applications. HAp has been tested many times as an artificial bone material especially as augmentation material in surgery work, or as a coating material on bio-inert implants materials and has shown excellent biocompatibility and bonding characteristics. Many implant materials has been made in the last three decades made of metals, alloys, ceramics and polymers etc.Thermoplastic polymers are popular due to their low density, good mechanical strength, and easy formability. In this study, the polymer is combined with ceramic fillers to make composite plate. The effect of various parameters in drilling of composite plate is evaluated using a Taguchi design. Machining of composite reveals the best optimum combination of parameters of vol. % of HAp, feed rate, speed and drill diameter to achieve minimum taper with maximum circularity at both entry and exit.

KEYWORDS: Biocompatibility, Circularity, Hydroxyapatite, Polypropylene, Thermoplastic, Taper.

I.INTRODUCTION

The development of advanced materials for biomedical applications is among the most important problems facing modern materials engineering. The greatest potential for bone substitution is shown by materials based on hydroxyapatite (HAp), $Ca_{10}(PO_4)_6(OH)_2$, which can develop tight bonding with bone tissue, exhibits osteo conductive behavior, is stable toward bio resorption, and has no adverse effects on the human organism. The biological behavior of HAp ceramics depends on many factors, in particular, on their chemical and phase composition, microstructure, pore size, and pore volume. When a HAp based ceramic is implanted, a fibrous tissue-free layer containing carbonated apatite forms on its surfaces and contributes to the bonding of the implant to the living bone, resulting in earlier implant stabilization and superior fixation of the implant to the surrounding tissues. Furthermore, several studies have shown that HAp or its derivatives can be exploited as a model compound to study bio mineralization in the human body. Recent studies have also shown that HAp particles inhibit the growth of many kinds of cancer cells. Currently, HAp is commonly the material of choice for various biomedical applications, e.g., as a replacement for bony and periodontal defects, alveolar ridge, middle ear implants, tissue engineering systems, drug delivery agent, dental material sand bioactive coating on metallic osseous implants. The general importance of HAp and its derivatives has also led to numerous non-medical industrial and technological applications, e.g., as a catalyst for chemical reactions such as the Michael-type addition and methane oxidation, host materials for lasers, fluorescence materials, ion conductors and gas sensors. Synthetic HAp may also be used in column chromatography for simple and rapid fractionation of proteins and nucleic acids. Moreover, it has been demonstrated that HAp presents very convenient qualities for water treatment processes and remediation of heavy metal contaminated soils.

Polypropylene (PP), also known as polypropene, is a thermoplastic polymer used in a wide variety of applications. An addition polymer made from the monomer propylene, it can be produced in a variety of structures giving rise to applications including packaging and labeling, textiles, plastic parts and reusable containers of various types, laboratory equipment, automotive components, and medical devices. It is a white, mechanically rugged material, and is resistant to many chemical solvents, bases and acids. Polypropylene is in many aspects similar to polyethylene, especially in solution behaviour and electrical properties. The methyl group improves mechanical properties and thermal resistance, although the chemical resistance decreases. The properties of polypropylene depend on the molecular weight and molecular weight distribution, crystallinity, type and proportion of comonomer (if used) and the isotacticity. In isotactic

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polypropylene, for example, the methyl groups are oriented on one side of the carbon backbone. This arrangement creates a greater degree of crystallinity and results in a stiffer material that is more resistant to creep than both atactic polypropylene and polyethylene.

Selection of machining parameter combinations for obtaining optimum circularity at both entry and exit and optimum hole taper is a challenging task owing to the presence of a large number of process variables. There is no perfect combination of parameters that can simultaneously result in higher circularity at both entry and exit with lower hole taper. The aim is to develop a strategy for predicting machining parameter settings for the generation of the maximum circularity at both entry and exit with minimum hole taper.

II. LITERATURE SURVEY

Pramanik et al.found that large pored HAp material has high strength and density. Neuendorf et al. advised that bulk polymer degradation after immersion in waterbased solutions results in a significant decrease in the mechanical properties of biodegradable copolymer. In early 1980s, researchers discovered self-setting calcium orthophosphate cements, which are a bioactive and biodegradable grafting material in the form of a powder and a liquid. Both phases after mixing form a viscous paste that after being implanted sets and hardens within the body as either calcium deficient hydroxyapatite (CDHA) or brushite. As CDHA and brushite are bio-compatible and bio-resorbable, calcium orthophosphate cements represent to be very promising materials for bone grafting applications.

Besides this, these cements possess an excellent osteo-conductivity, molding capabilities and easy manipulation. The first in vivo use of calcium orthophosphates was performed in 1920; that time the researchers implanted tri-calcium phosphate (TCP) into animals to test its efficacy as a bone substitute. In 1951, the first time HAp was implanted in animals. In the year 1970s, various other calcium orthophosphates like α-TCP, ß-TCP, amorphous calcium phosphate, calcium deficient hydroxyapatite (CDHA), Fluorapatite, Tetra-calcium phosphate (TTCP) etc. were synthesized, characterized, investigated, and tried in medicine.

Moreover, calcium orthophosphate cements can be injected directly into fractures and bone defects, where they intimately adapt to the bone cavity regardless its shape. In besides of this, they possess sufficient compressive strengths, non-cytotoxic in nature, create chemical bonds to host bones, and have both the chemical composition and X-Ray Diffraction (XRD) patterns similar to those of human bone. Finally, but yet importantly, they are osteotransductive i.e., after implantation calcium orthophosphate cements are replaced by a new bone tissue. Therefore, the discovery of self-setting calcium orthophosphate cements was a significant step forward in the field of bio-ceramics for bone regeneration. Jagur et al. found that the biocompatible and biodegradable collapsible polymeric stents, as wellas polymeric materials are used for protective coatings of metallic stents and reservoirs of drugs, preventing restenosis and other post-operative complications that may occur after insertion of a stent.

Garnett et al. proposed that in vitro, the organic molecules of serum play a more important role in controlling the nucleation and growth of HAp than the inhibitory inorganic ions while in vivo, organic and inorganic molecules coexist both in serum and in bone mineral and so their effects also depends upon interactions with one another as well as interactions with HApcrystals. Coombes et al. informed that fibre reinforced polymers offer opportunities for achieving good structural properties in thin material sections for guiding and supporting bone growth.

Wettergreen et al. advised that the combination of computer-aided design (CAD) with imaging techniques helps to create patient-specific tissue constructs and to enumerate potential methods for the reconstruction of an entire human vertebral body. Rai et al. suggests that bone material shows interactions of different component with change in temperature. Ramakrishna et al. informs that the polymer composite biomaterials are particularly attractive because of their tailorable manufacturing processes, and properties comparable to those of the host tissues.

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III. METHODOLOGY AND EXPERIMENTAL DETAILS

Analytical grade Calcium Hydroxide ($Ca(OH)_2$) powder (Merck, 96%) and Orthophosphoric (H_3PO_4) acid (Merck, 85%) were weighted at molar ratio of Ca/P=1.67 to prepare HAp powder as homogenous solution. In this method, initially, ten grams of Calcium Hydroxide is weighted in a weighing machine (Mettler Toledo, 0.01 g accuracy). It is mixed with water about 40 times. The solution is stirred by a magnetic stirrer (RemiEquipmentsPvt. Ltd.) at 50° C to 60° C for 3 to 4 hours. Then, Ortho Phosphoric acid is mixed with the solution at the rate of 30 drops/minute through a burette and continuously stirred but heat input to the solution is stopped. Care is taken to reach at pH value of the solution at 8 to 10. If pH value of the solution drops below the threshold values, Ammonia solution may be added to increase the pH value. After 5 to 6 hours, magnetic stirrer is stopped and the mixer is keep for 12 hours at room temperature. Then, precipitations are collected using a filter paper. The HAp precipitations are dried at 80ºC in an oven and calcined at 850ºC for 2 to 3 hours in muffle furnace.

Hand lay-up technique is the simplest method of composite processing. The infrastructural requirement for this method is also minimal. The processing steps are quite simple. First of all, a release gel is sprayed on the mold surface to avoid the sticking of polymer to the surface. Thin plastic sheets are used at the top and bottom of the mold plate to get good surface finish of the product. Reinforcement in the form of woven mats or chopped strand mats are cut as per the mold size and placed at the surface of mold after perspex sheet. Then polymer in liquid form is mixed thoroughly in suitable proportion with a prescribed hardner (curing agent) and poured onto the surface of mat already placed in the mold. The polymer is uniformly spread with the help of brush. Second layer of mat is then placed on the polymer surface and a roller is moved with a mild pressure on the mat-polymer layer to remove any air trapped as well as the excess polymer present. The process is repeated for each layer of polymer and mat, till the required layers are stacked. After placing the plastic sheet, release gel is sprayed on the inner surface of the top mold plate which is then kept on the stacked layers and the pressure is applied. After curing either at room temperature or at some specific temperature, mold is opened and the developed composite part is taken out and further processed. The time of curing depends on type of polymer used for composite processing.

For obtaining optimum circularity at both entry and exit with optimum hole taper, some machining parameters are considered with their levels. Four parameters were chosen as inputs i.e., HAp vol. %, feed rate, speed, and drill diameter. Experiments were carried out using Taguchi mixed-level design L16 orthogonal array combinations of these factors and their different levels. The experiments were performed on radial drilling machine. In this experiment, analysis of the effect of different parameter settings on circularity at entry and exit and hole taper on HAp-PP composite is carried out. The responses considered were circularity at entry, circularity at exit, and hole taper of the holes. The thickness of the job sample is measured at different sections using a digital verniercaliper having least count of 0.01mm.The diameters of the drilled holes were in millimeter range and the holes were drilled without any relative motion between the job and the work piece. After completion of the experiment, microscopic views of the drilled holes at both top (entry) and bottom (exit) were taken with the help of an optical measuring microscope. The circularity at hole entry and hole exit, and the diameter of the drilled hole at entry and exit, were measured by analyzing the microscopic views of holes with image analysis software. The circularity at both entry and exit is measured by using the ratio of minimum to maximum diameters of the hole. After measuring the entrance diameter and exit diameter of the hole, the hole taper is calculated.

IV. EXPERIMENTAL RESULTS

Microstructure examinations were carried out using scanning electron microscopy (SEM). Specifically Field Emission Scanning Electron Microscope JEOL SEM – JSM 6610LV, which can provide high-resolution performance (up to 3 nm), were used to detect the presence of nanoparticle in the bulk sample. Kevex energy dispersive x-ray spectroscopy (EDS) system and X-ray mapping were also utilized to characterize chemical elements and their distribution. Since the average size of the nano-particles is less than 60 nm, it is very difficult to use EDS spot analysis on a single particle due to the limitation of the e-beam resolution in this instrument.

For XRD studies of HAp composite (Model: 2036E201; Rigaku, Ultima IV, Japan) the 150x60mm solid specimens are prepared for the characterization purpose. The surface of the composite sample must be in plane with the sample holder. The XRD is power on and the voltage and the current values are set to standard values i.e. 40kV and 20mA. Now the

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XRD pattern is generated with a speed of 2 deg per minute and the pattern is generated from 2 deg to 90 deg. The generated XRD pattern is now analyzed with JADE 7.0 Elevation software for structural information. From this we can obtain crystallinity of the composite sample, phase identification, crystallite size of the phase identified, planes orientation, crystal structure of the phase and residual strains of the crystals. To identify the phase and crystal structure of a given material, XRD is used. Through XRD technique, we also know the form of a given material for whether it is pure or not. The experimental data calculated for Taper, Circularity at entry and Circularity at exit are shown in Table 1.

Table 1 Experimental Data for Taper, Circularity at entry and circularity at exit

HAp vol. % (%)	Feed Rate (mm/min)	Speed (rpm)	Drill Diameter (mm)	Taper (radian)	Circularity at entry (mm)	Circularity at exit (mm)
10	50	750	5	0.0033	0.9642	0.8217
10	100	1500	5	0.0099	0.8415	0.7821
10	150	2250	6	0.0066	0.8495	0.8388
10	200	3000	6	0.0066	0.8666	0.8544
20	50	1500	6	0.005	0.8356	0.8084
20	100	750	6	0.0017	0.845	0.835
20	150	3000	5	0.0067	0.8352	0.8314
20	200	2250	5	0.0017	0.8554	0.8502
30	50	2250	5	0.0032	0.8514	0.8166
30	100	3000	5	0.008	0.8379	0.8128
30	150	750	6	0.0048	0.8383	0.8402
30	200	1500	6	0.0016	0.8374	0.8316
40	50	3000	6	0.011	0.8238	0.8
40	100	2250	6	0.0047	0.8272	0.8125
40	150	1500	5	0.0015	0.8187	0.8238
40	200	750	5	0.0031	0.8474	0.8352

To optimize the multiple responses into a single response to get the optimum parametric combination, various methods are used. To get the best optimum parametric condition in less time, we used Weighted Principal Component Method. This method takes few times and gives the optimum combination value simultaneously. The process parameters need to be determined in such a way that they collectively optimize more than one response simultaneously. To address this issue, effect of important process parameters viz., HAp vol. %, feed rate, speed, and drill diameter have been studied. The responses considered in this study are taper, circularity at entry and circularity at exit. The multiple responses are converted into a single response using principal component analysis (PCA) so that influence of correlation among the responses can be eliminated. Resulting single response is nothing but the weighted sum of three principal components that explain hundred percent of variation. The experiments have been conducted in accordance with Taguchi's orthogonal array to reduce the experimental runs.

Let Y_i be the normalized value of the i^{th} response, for i =1, 2,…p. To compute PCA, k (k≤p) components will be obtained to explain variance in the p responses. Principal components are independent (uncorrelated) of each other. Simultaneously, the explained variance of each principal component for the total variance of the responses is also obtained. The formed j principal component is a linear combination $Z_j = \sum_{i=1}^{p} a_{ji} Y_i$ for j = 1, 2,…k subjected to $\sum_{i=1}^{p} a_{ji}^2 = 1$; also, the coefficient a_{ji} is called eigenvector.

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In weighted principal component method, all principal components will be used; thus the explained variance can be completely explained in all responses. Second, because different principal components have their own variance to account for the total variance, the variance of each principal component is regarded as the weight. Because these principal components are independent to each other (which means that these principal components are in an additive model), the multi-response performance index (MPI) is MPI = $\sum_{j=1}^{k} W_j Z_j$, where Wjis the weight of *j*th principal components. The larger the MPI is the higher the quality. Finally, with the application of ANOVA (Analysis of Variance), significant factors in this quality index and their contribution percentage for total variation in MPI can be obtained. First, three responses are normalized to avoid the scaling effect. Three principal components are obtained using PCA. It is evident from Table 2 that principal component 1 (PC1) explains 54.264 % of variance, (PC2) explains 30.230 % and (PC3) explains 15.506 % of total variance. The calculations for principal components are done according to the following relation:-

PC1 = (-.845 × Taper) + (.223 × Circularity at entry) + (.485 × Circularity at exit)
PC2 = (.459 × Taper) + (.888 × Circularity at entry) + (.019 × Circularity at exit)
PC3 = (.838 × Taper) + (-.261 × Circularity at entry) + (.479 × Circularity at exit)

Table 2 Explained variation and Eigen vector

Principal Component	Eigen value	Explained variation (%)	Cumulative variation (%)	Eigen vector (Taper, Circularity at entry, Circularity at exit)
PC1	1.628	54.264	54.264	[-0.845, 0.223, 0.485]
PC2	.907	30.230	84.494	[0.459, 0.888, 0.019]
PC3	.465	15.506	100.000	[0.838, -0.261, 0.479]

The relation for weighted principal component, MPI, is given by

MPI = (0.5426 × PC1) + (0.3023 × PC2) + (0.1550 × PC3)

Table 3 shows the principal components and MPI values with normalized values of responses. ANOVA Table for MPI and response Table for MPI is shown in Table 4 and Table 5 respectively.

Table 3 Principal Components and MPI

Expt.	Normalized Value			Principal Components			MPI
No.	Taper	Circularity at entry	circularity at exit	PC1	PC2	PC3	
1	0.3	1	0.96173	0.4359	1.0439	0.451	0.6219
2	0.9	0.87274	0.91538	-0.1219	1.2054	0.9648	0.4477
3	0.6	0.88104	0.98174	0.1655	1.0763	0.743	0.5303
4	0.6	0.89878	1	0.1784	1.0924	0.7472	0.5428
5	0.45455	0.86663	0.94616	0.268	0.9961	0.6078	0.5407
6	0.15455	0.87637	0.97729	0.5388	0.8676	0.3688	0.6117
7	0.60909	0.86621	0.97308	0.1504	1.0672	0.7503	0.5205
8	0.15455	0.88716	0.99508	0.549	0.8775	0.3745	0.6212
9	0.29091	0.88301	0.95576	0.4146	0.9357	0.471	0.5808
10	0.72727	0.86901	0.95131	0.0406	1.1235	0.8382	0.4915
11	0.43636	0.86943	0.98338	0.3021	0.9909	0.6097	0.5579
12	0.14545	0.86849	0.97331	0.5428	0.8563	0.3614	0.6093
13	1	0.85439	0.93633	-0.2003	1.2354	1.0635	0.4296
14	0.42727	0.85791	0.95096	0.2915	0.9759	0.5895	0.5445
15	0.13636	0.8491	0.96419	0.5417	0.8348	0.3544	0.6012
16	0.28182	0.87886	0.97753	0.4319	0.9282	0.475	0.5885

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Table 4 ANOVA Table for MPI

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% contribution
Vol. %	3	0.003652	0.003652	0.001217	0.35	0.794	10.4817
Feed Rate	3	0.009386	0.009386	0.003129	0.89	0.506	26.9387
Speed	3	0.021095	0.021095	0.007032	2.00	0.232	60.5447
Drill Diameter	1	0.000709	0.000709	0.000709	0.20	0.672	2.0349
Total	10	0.034842	0.034842	-	-	-	100

Here we get the S value of 0.0592371, R-Sq value of 66.51% and R-Sq (adj) value of 0.00 %.

Table 5 Response Table for MPI

Level	Vol. %	Feed Rate	Speed	Drill Diameter
1	0.5357	0.5433	0.5950	0.5592
2	0.5735	0.5239	0.5497	0.5459
3	0.5599	0.5525	0.5692	-
4	0.5410	0.5904	0.4961	-
Delta	0.0378	0.0666	0.0989	0.0133
Rank	3	2	1	4

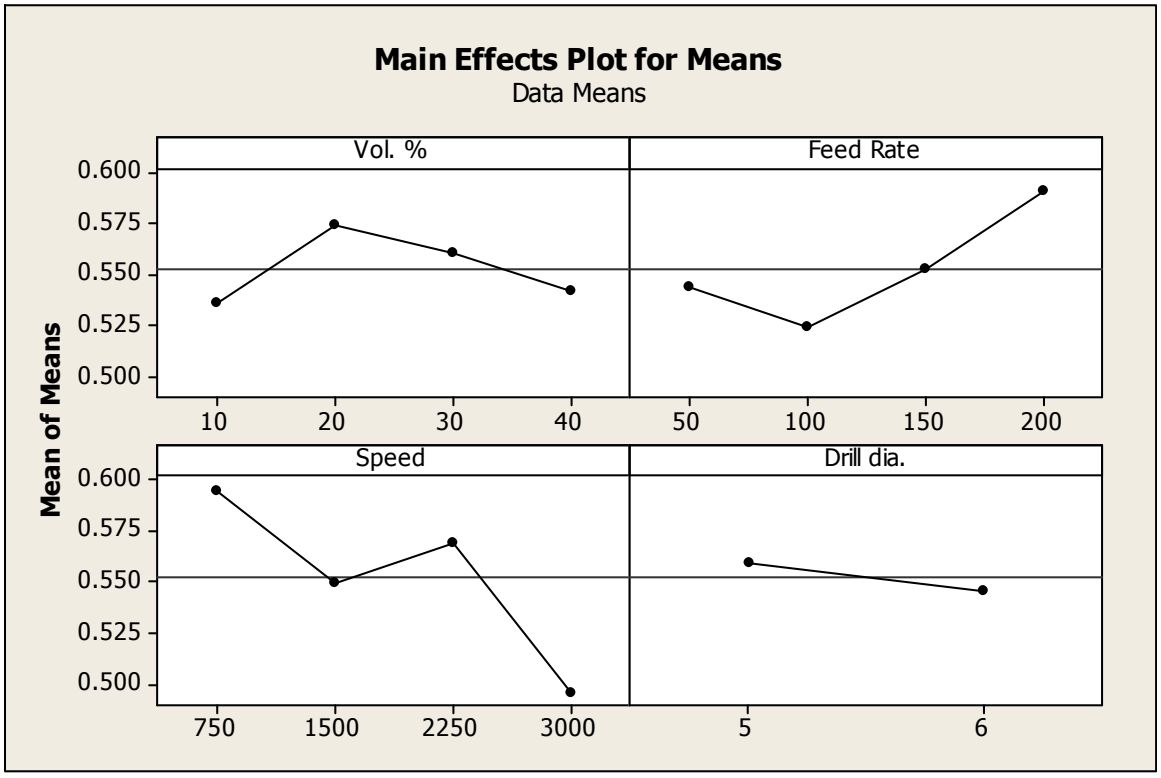

Figure 1 Main effects plot for means of MPI

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Hence, main effects plot for means of MPI (Figure 1) shows the optimal parametric combination is $A_2B_4C_1D_1$, which shows the higher-the-better (HTB) value. It means that when feed rate of 200 mm/min, Speed of 750 rpm and 5mm drill diameter is machined or drilled in 20 vol. % of HAp sample, then we get minimum taper with maximum circularity at both entry and exit.

V.CONCLUSION

It has been observed that the combination of HAp-PP composite gives more satisfactory results than the combination of other HAp composite. These results suggest that the bioactive HAp-PP composite have the potential for use as an alternative material for load-bearing orthopaedic applications. Good mechanical compatibility of HAp-PP thermoplastic bio-composite with those of the natural bone is evident. The crystal size is calculated for dry HAp is 33.681 nm whereas the crystal size for sintered HAp is calculated at 850°C is 40.413 nm because of grain size is increasing with respect to heating. When feed rate of 200 mm/min, speed of 750 rpm and 5mm drill diameter is machined or drilled in 20 vol. % of HAp sample, then we get minimum taper with maximum circularity at both entry and exit.

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AUTHOR'S BIOGRAPHY

1.Dr. Amit Kumar Meharis an Associate Professor in the Department of Mechanical Engineering, Raghu Engineering College (REC) (Autonomous), Vishakhapatnam, Andhra Pradesh, INDIA. He has 3 years of industrial experience, 4 years of teaching experience, and 6 years of research experience in his field. He has 3 reputed international journals, 10 national and international conferences, and one edited book chapter. He has 10 foreign professional memberships. He was born on 24th January 1984 in Raipur, (Chhattisgarh), INDIA. He did B.E. in Mechanical Engineering from K.I.T., (Government College), Raigarh (Chhattisgarh), INDIA in the year 2006. He did both M. Tech. and Ph.D. in Mechanical Engineering from N.I.T., Rourkela (Odisha), INDIA in the year 2011 and 2017 respectively. His area of research interest is related to biomaterials, composite materials, tribology, machining, optimization techniques, soft computing etc.

2. Mr.SreekanthKotniis an Associate Professor in the Department of Mechanical Engineering, Raghu Engineering College (REC) (Autonomous), Vishakhapatnam, Andhra Pradesh, INDIA. He has 2 years of industrial experience and 10 years of teaching experience. He has one reputed international journal, one professional membership and also heattended various seminars, conferences and workshops in his field in INDIA. He was born on 10th July 1973 in Vishakhapatnam, Andhra Pradesh, INDIA. He did both B.Tech. andM.Tech. in Metallurgical Engineering from N.I.T., Warangal, Andhra Pradesh, INDIA with distinction in the year 1999 and 2001 respectively. His area of research interest is related tobiomaterials, composite materials, machining, optimization techniques, etc.