



## Designed and fabricated a low-cost E-Spun experimental tool for polymer processing

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Received: 4 November 2020; Accepted: 21 March 2021

Nanoscale fiber processing involves advanced technology that is challenging to use and setup in the laboratory at low cost. To deal with such a dilemma, this experimental setup explores the notion that would stimulate researchers' curiosity about the polymer processing electrospinning tool. This study has presented a simple, precise, portable, cost-effective, and economical electrospinning setup as a learning resource that is well-suited, laid-back and requires intangible knowledge of fabricating and installation. This proposed instrument consists of two main units syringe pump and stationary collector. A medical grade syringe has been used as a pump sprayer to spray the fibers. Super arc 1000 kV high-voltage generator has been used for electrospinning setup. A flat copper plate has been used as an electrode to secure the fibers of the prepared viscous PVA solution. A set of experiments have been carried out to evaluate the performance of the proposed and described design. Surface morphology results have been examined by SEM at different magnifications, confirming the formation of fibers and the diameter of PVA fibers is 0.926  $\mu\text{m}$ . XRD studies have been used to verify that the crystallinity increases by 23.22% to 25.54%, which shows a gradual optimization in mechanical properties.

**Keywords:** E-Spun, Syringe pump, Cost-effective, PVA fibers

### 1 Introduction

In recent years, natural fibers and reinforced fiber composites have received much attention because of their lightweight, durable, flammable, non-toxic properties, low cost, and corrosive properties. Much research work has been done around the world on the use of natural fibers as stabilizers in the preparation of various chemical reactions in a variety of ways. Electrospinning (e-spun) technology was first introduced in 1934 and has attracted physicists and materials scientists worldwide<sup>1</sup>. Nanofibers preparation methodologies include tensile method<sup>2,3</sup> template synthesis process<sup>2,4</sup> self-assembly approach<sup>2,5</sup> and ultrasound process<sup>2,6</sup> including electrospinning. The e-spun method has become simpler and easier to prepare the ultra-fine fibers<sup>7</sup> by using a high-voltage power supply, with an average diameter of several micrometers to several hundreds of nanometers<sup>8</sup>.

As the product size of the polymer fiber is reduced from micrometer to nanometer, a number of impressive features appear, such as a large surface area to a large volume high flexibility in surface

functionality and high mechanical strength. Such outstanding properties ensure that polymer nanofibers are the ideal choice for many outstanding applications in various fields such as tissue regeneration<sup>9,10</sup> and drug delivery<sup>2,11</sup> energy storage<sup>12,13</sup> biosensor<sup>14,15</sup> composites<sup>16</sup> biomaterials<sup>17</sup> and wound dressing<sup>18</sup>. However, the performance of the e-spun depends on parameters such as applied voltage<sup>14</sup> and polymer concentration<sup>19</sup> which are affected by fiber width and production rate<sup>20</sup>.

Water-based e-spun is key to the success of many areas such as biomedical use<sup>17</sup> agricultural use<sup>21</sup>, and wound treating<sup>9</sup>. This method can be used for many different polymers such as water-soluble polyvinyl alcohol (PVA)<sup>22</sup>, polyvinyl pyrrolidone (PVP)<sup>4</sup>, polyethylene glycol (PEG)<sup>14</sup>. In particular, water-soluble PVA has high mechanical, chemical, and biological properties<sup>23</sup>. This e-spun is a unique type of process wherein the polymer solution and melting are spun and disturbed by the operating conditions of high-voltage power systems<sup>20</sup>. Solution and processing parameters usually include viscosity<sup>14</sup>, molecular weight<sup>24</sup>, polymer concentration<sup>2</sup>, the tip of a needle and collector's distance<sup>25</sup>, conductivity<sup>7</sup>,

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device including ambient conditions, significantly affecting morphology of fibers<sup>16</sup>. The desired properties for various applications can be engineered by modifying the above parameters. In e-spin, a polymer solution that is sprayed through a needle is applied with high voltage<sup>26</sup>.

A drop is shaped at the edge of a needle, which is again deformed into the Taylor cone on the electrical region, and a jet is expelled from the Taylor cone as the electrostatic forces on the drop exceed the surface tension<sup>24,26</sup>. While the solvent evaporates immediately from the jet, the jet is subjected to greater static repulsion and instability that contribute to waving of the fiber, which enables very small/thin fibers to dry on the collector surface plate<sup>2,26</sup>. Figure 1 shows the schematic representation of the Taylor cone (needle) and spray. Routinely, fixed aluminum foil is used to process electro-spun liquids in a collector face plate<sup>19</sup>.

Certain types of collectors such as rotating cylinder<sup>2,27</sup>, rotating circle<sup>24</sup>, wire mesh<sup>28</sup>, pin and parallel<sup>29</sup>, or grid bar are often used to develop unidirectional and bidirectional nanofibers<sup>30</sup>. The plate can form non-woven fibers, whereas this part of the rotation may produce non-woven and aligned fibers. It is inferred from an overview of literature that many research teams are working on the construction

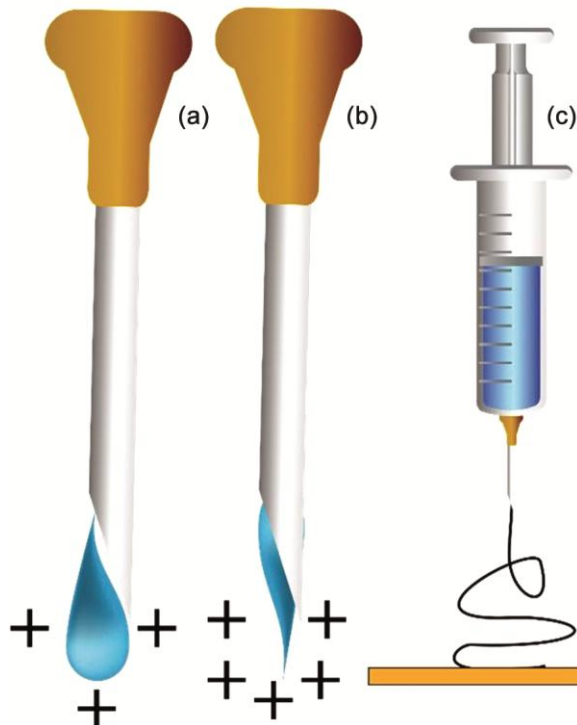


Fig. 1 — Visualization of the development Taylor cone and spray (a) Droplet in tiny electric field, (b) creation of Taylor cone in high electric field, and (c) e-spun in high electric field.

of electrospinning. The Universidad Nacional Autónoma de Mexico's Materials Research Institute has designed and built several electrospinning devices<sup>31</sup>. The design reported in the study is the basis and an important motivator of the present investigation. Currently, two standard electrospinning combinations are available in the market<sup>14</sup>. The proposed design is aimed at transforming those instruments that are considered an essential tool in all research. A horizontal electrospinning framework is planned, and then designed and built using some resources available in the market and online. PVA polymer fibers have been processed from the home-made e-spin research station and exposed to morphological characterization.

## 2 Materials and Methods

### 2.1 Design and fabrication

The primary design and construction of the electrospinning setup are shown in Fig. 2. The complete mechanical system comes with a slide stroke CNC Z Axis linear motion actuator (Machifit 150mm,T8-Z60), which is made up of stainless steel (silver and black color) with slide stroke of 150mm, motor steps of 800 steps/mm, rotating screw pitch of 2 mm, along with 12-V power and 1.7A current rating. Furthermore, the dimensions of stepper motor are  $41 \times 170 \times 67$  mm along with the maximum pull capacity of 4.5 kg and it is positioned on a vibration-resistant table. In addition, the dimension of each part is shown in Fig. 3. M3 screws were used for adjustment and positioning alignment. This was found to be required for components in development. However, further investigation is needed to examine the details of components used in the setup. A combination of the L293D motor control shield was purchased from M/s. MH Electronics and Robototix

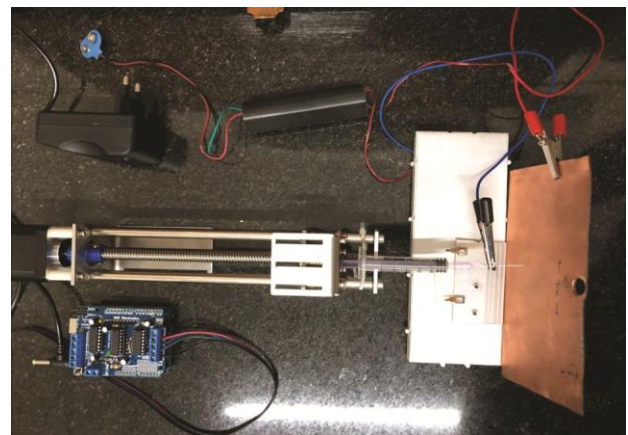


Fig. 2 — Snapshots of low-cost homemade electro-spinning setup.

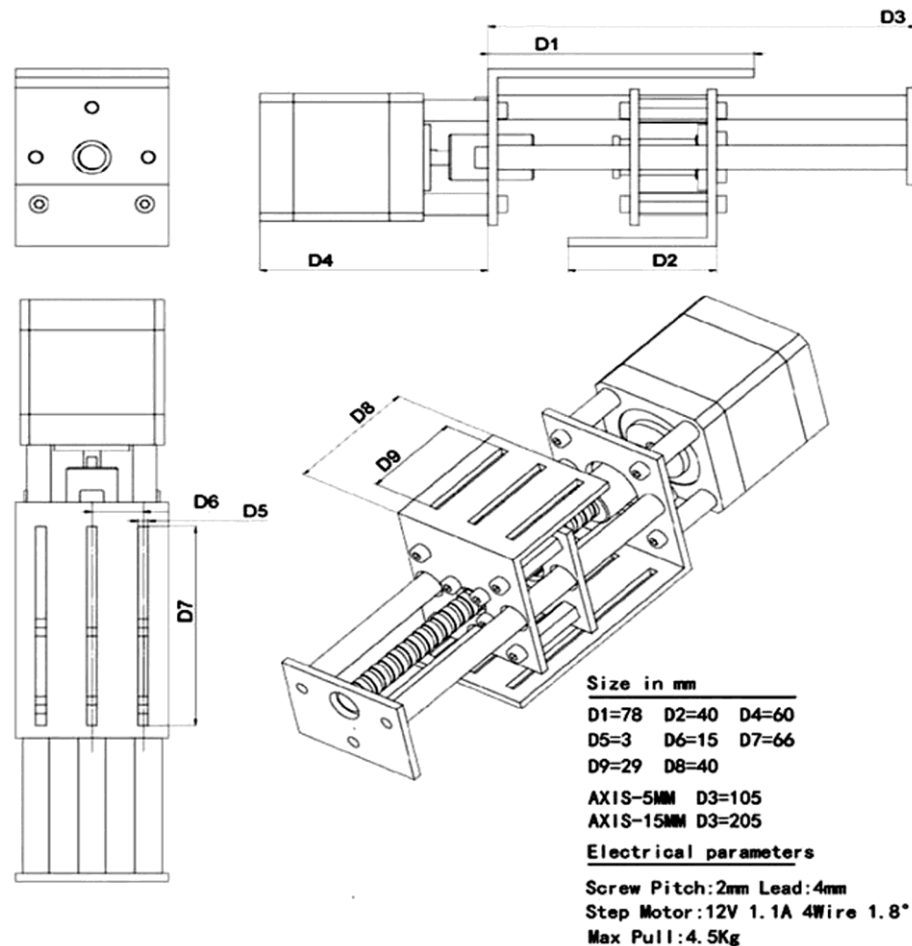


Fig. 3 — Dimension of Machifit 150mm, T8-Z60 in details.

Arduino Uno 328P micro-controller board was used and uploaded the code was uploaded to Arduino, which is used to control the stepper motor using Arduino software (IDE).

An adapter (12V) was used as a power supply unit on the board as stepper motor takes multiple pulses to reach optimal exact/targeted position. On the other hand, high voltage supply (Super Arc 1000 kV high-voltage generator) was used to generate/control the electrospinning process. A high-voltage transformer inverter pulse module with technical specification of input voltage DC 3.7-10.4 V input current of 2–5 A with size of 88 × 26 mm, the high voltage line length about 100 mm and discharging distance of 10–20mm with 100mm input and output cable length (red for positive terminal) was used. This high-performance, low-volume, simplified peripheral circuit and high-voltage module can be operated using DC power supply/battery. A solution sprayer injection pump was constructed using a medical-grade syringe (2ml) with a regulated/controlled flow rate. The flow rate of the

syringe pump was adjusted by coding in Arduino board. Nylon cable was used for binding (SL-100; Surelock) the syringe holder with size of 104×2.5 mm. The surface of the collector must be a strong conductor like copper/aluminum. Here, a stationary copper collector was used to collect the fibers from syringe needle tip with dimensions of 150 × 80 × 0.2 mm and a rectangular copper plate was used (Fig. 4).

## 2.2 Working

Assembling this type of motor is very convenient and requires information on the four terminals (black, green, blue, and red) of stepper motor connected to M3 and M4 of the motor control board. In designing of the instrument, the 12 V adapter was upgraded for supplying power to the coded microcontroller board using Arduino software. For high-voltage power supply, a 9V Hi-Watt battery with a battery clip connector was used as input (Fig. 2). A mini two-pin SPST on/off switch was installed to control the source board. A medical-grade 2ml syringe was filled with a

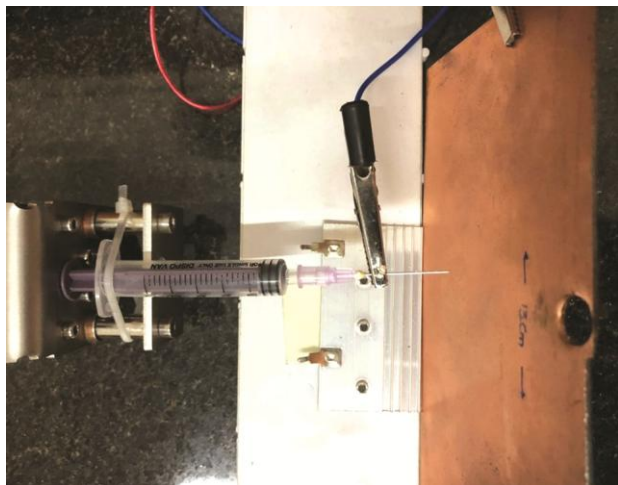


Fig. 4 — Zoom view of syringe needle and collector plate.

selected polymer and placed in the syringe-holder where the nylon tie acted as a holder. The positive terminal of the high-voltage module was connected to the stationary collector (copper plate) and the negative terminal to the tip of the needle or vice versa. Distance was fixed between the needle and the stationary collector (~2 cm). After rechecking the wires/inspecting the components, we switched on the high-voltage power supply and waited for a while to build the electric potential between the collector and syringe, expecting formation of the Taylor cone. Further, we switched on the power supply for microcontroller board using the 12V adapter. Within a fraction of second the stepper motor started to rotate and pulled the syringe in the perpendicular direction of the stationary collector spraying the fibers. This method is both simple and sufficient regarding formation of fibers, which were collected in the run time of 5 min and subjected for characterization. Each component that accompanied the design and development of the electrospinning equipment was purchased from Bangood and local stores of Dharwad (15.4589° N, 75.0078° E) and Davangere (14.4644° N, 75.9218° E).

### 2.3 Validation–Production of PVA fibers

To validate this, an experiment setup was used for producing nanofibers. PVA was purchased from M/s. HI-Media Laboratories Pvt Ltd., Mumbai. PVA is soluble in water with average molecular weight of 160,000 and degree of hydrolysis 86.5–89.0 mol%. Then, 1.5g PVA was dissolved in 50 ml deionized water and the prepared solution was continuously stirred at 40°C for 24 h. After complete dissolution, the solution was stored overnight to eliminate

bubbles. The PVA solution was injected into a medical-grade syringe (2ml) without any bubbles and positioned in the syringe-holder. It was even further tested for a length of 2 cm from the needle tip. In this experiment, the Super Arc 1000 kV high-voltage generator operating at 9 V was used for the generation of high-voltage supply output. Fibrous samples were left on the electrospinning device (collector) for 5 mins. The PVA fiber surface morphology and structure were successfully tested using a scanning electron microscope (SEM) and X-ray diffraction (XRD) method. The SEM samples were captured at different magnifications. The processed samples were characterized by SEM (model JSM-IT500 In Touch Scope; JEOL), which ensured the visibility of the fiber. The prepared polymer fiber and PVA film's crystallinity index was studied using powder XRD (M/s Rigaku X-ray Diffractometer, model Smart Lab SE with a Cu K  $\alpha$  source at 2 $\theta$ , scan range of 5°–80°, step width of 0.02°, and scan speed of 5.00°/min)

### 3 Results and Discussion

Figure 5 shows the SEM images of the prepared PVA nanofibers along with random fiber formation of pure PVA, porosity, diameter, and fiber diameter consistency. The data clearly show formation of fiber along with bead-like fiber structure. Research findings have suggested that lower solution concentrations develop fibers with beads and the shape is modified between beaded fibers and smooth and uniform fibers through improved solution concentration<sup>9,14,23</sup>. Optimal condition was selected to study the effects for the minimal distance (2cm) from the collector. Based on the results, the following observations were made: the fiber diameter is indeed one of the main electrospinning quantities. Average fiber diameter was computed using software Image J from the SEM micrographs of Fig. 5(a) of 20 $\mu$ m resolution with 5 kV accelerating voltage. The diameter was found to be 0.926  $\mu$ m for 100 different fibers, which was estimated from the first plane and measured from the Gaussian curve fitting diameter distribution histogram, as shown in Fig. 5(b). The percentage of porosity was found to be 60%.

For comparison studies, we began with the preparation of PVA films using solution casting method. In this method, we added 3g PVA to 60ml deionized water and kept for 24-h stirring at 40°C for complete dissolution, and the obtained product was named as pure PVA. XRD profiles (Fig. 6) were used

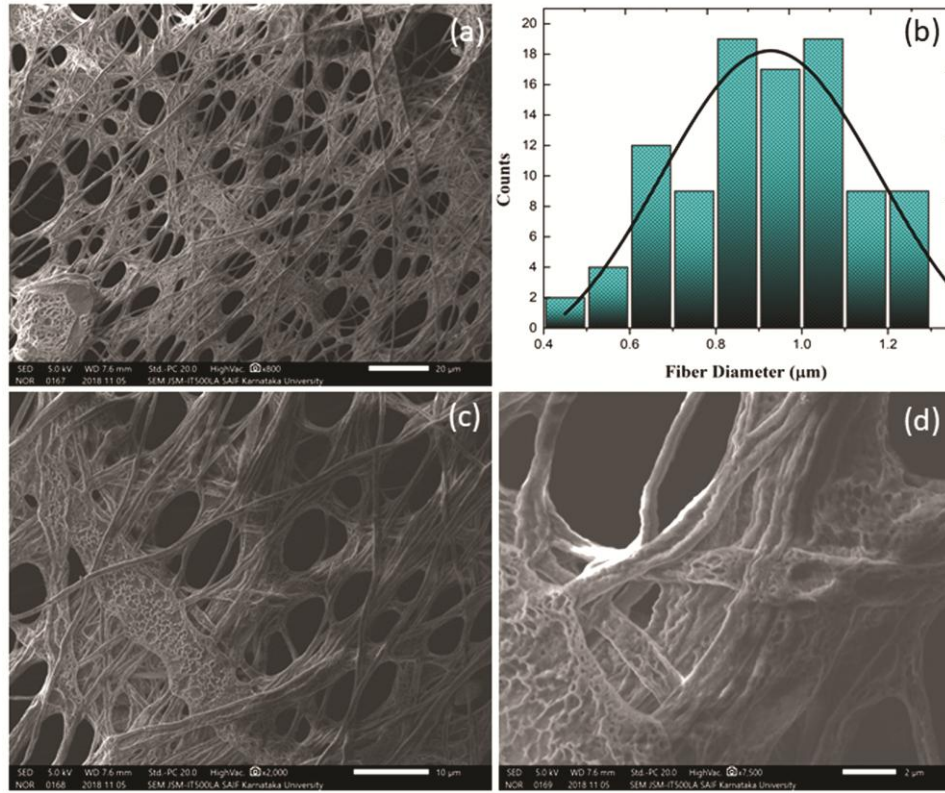


Fig. 5 — (a), (c), (d) Sem visuals of prepared PVA polymer fiber at different magnification and (b) distribution histogram and Gaussian curve fitting for prepared fiber diameter.

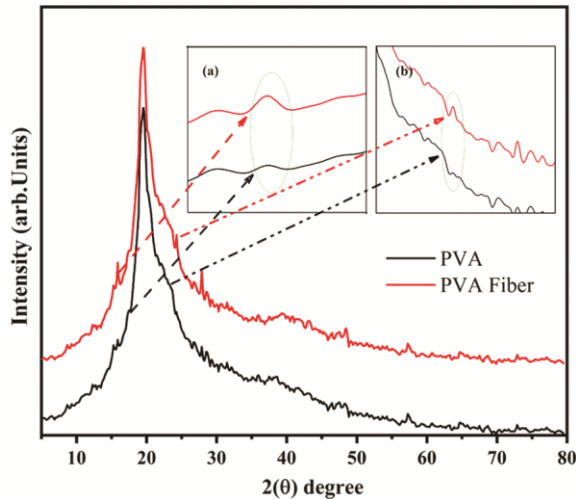


Fig. 6 — XRD Patterns for prepared PVA fiber and solution casted PVA film (a), (b) zoom view to verify the crystallinity in both polymers.

to describe PVA and PVA fiber crystallinity. Noticeable diffraction peaks were found around 19.46° in both. There was a significant correlation to the (101) plane semicrystalline structure of PVA and PVA fibers distributions. Good agreement was found, when comparing results from this work against published

data<sup>32,33</sup>. Each diffraction profile was processed through a gaussian function peak-fit in Origin<sup>®</sup> 9 to acquire area of large amorphous halo and sharp peaks from the crystalline peaks. The crystallinity index for PVA and PVA fiber was calculated using the method reported by Pardo *et al.*<sup>34</sup>, as described in Eq. (1), and it is found to be 23.22% and 25.54%.

$$CI = \frac{A_c}{A_c + A_a} \quad \dots(1)$$

where,  $A_c$  is the region under crystalline spectra and  $A_a$  is the area of amorphous hollows.

Owing to excessive voltage potential, a majority of intrachain or interchain hydrogen bonds were developed during electrospinning, contributing to higher crystallinity<sup>35</sup>. The change in crystallinity was noticeable for PVA fiber at 15.85° and 24.24° due to electrospinning and stretching<sup>36</sup> of PVA, as shown in Fig. 6(a and b). Interestingly, due to their ordered structure, higher crystalline polymers typically have higher mechanical properties<sup>37,38</sup>. Studies have shown that an improvement in crystallinity can be achieved by electrospinning. In addition, the

method helped to optimize the microstructure as well as the physical and mechanical properties of fibrous materials.

#### 4 Conclusion

In this study, this process/setup has produced good results for fiber production using low-cost electrospinning designed and fabricated by easily accessible materials. This has 95% better value for money than the setup available in the market. SEM interpretation has confirmed morphological visual content and displayed the presence of PVA fibers with beads. The results have shown that crystallinity index computed using XRD spectrum for solution-casted film is 23.22%. Further, it can be enhanced by the designed electrospinning method, which increases the value to 25.54% for processed PVA polymer fiber. After rigorous usage of the device, it has been found that overheating is the limitation of this designed work. Future studies should focus on improving energy efficiency of high-voltage power supply. While this device has been beneficial for didactic functions, it can be effectively customized for proper research purposes. As a result, developed as well as underdeveloped laboratories with limited resources can be used for the teaching and learning process with this sharp, precise, portable, low-cost tool discussed in this study.

#### Acknowledgment

The authors acknowledge the support provided by the UGC-JRF/SRF (518772/Dec 2015). Chetan Chavan thank to the UGC, New Delhi. Soumya S Bulla acknowledges the monetary support granted by Karnatak University in the form of University Research Studentship including Department of Science and Technology, Karnataka Government, India, for the award of KSTePS Research Fellowship (DST/KSTePS/PhD Fellowship/PHY-04:2018-19). This work is also jointly supported by Davangere University. The experimental facilities offered by the USIC (University Scientific Instrumentation Centre) and SAIF (Sophisticated Analytical Instrumentation Facilities), Karnataka University, Dharwad, are appreciated. The authors also acknowledge Science and Engineering Research Board, Department of Science and Technology, Government of India, New Delhi, for the research project (SB/EMEQ-089/2013). The authors are grateful to UGC, New Delhi, for

providing financial assistance under SAP-CAS Phase-II program (F.530/9/CAS-II/2015(SAP-I).

The authors are also thankful to Karnataka State Higher Education Council, Govt. Of Karnataka for RUSA 1.0 grant (KSHEC/254/KUD/15-161544) at K.U. Dharwad.

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