# CHAPTER 4
# MATERIALS AND METHODS

## 4.1 MATERIALS, CONSUMABLES AND ACCESSORIES

Reinforcement and matrix are considered as basic material to manufacture composite laminate. Along with these, consumables and accessories are used to prepare the laminate with VARTM process. Consumables are those which are consumed during experiments and cannot be reusable, while accessories can be reused.

### 4.1.1 Materials

To manufacture composite laminates by VARTM process jute and glass fabric have been used as reinforcement. Jute is procured from Calcutta and glass fabric is procured from local supplier. General purpose polyester resin is used as matrix and is purchased from local supplier. Details of material are provided in Table 4.1.

**Table 4.1**.Materials

| Sr. No. | Item | Technical Specification |
|---|---|---|
| 1 | Jute | Jute fabric natural (untreated)<br>51" width x 290 GSM TPI:- 15x14 |
| 2 | Hollow Glass fabric | 211 GSM, 0.24 mm thickness,<br>Twill weave |
| 3 | Glass fabric 400 GSM | 400 GSM, 0.45 mm thickness,<br>Plain weave |
| 4 | Glass fabric 200 GSM | 200 GSM, 0.17 mm thickness,<br>Plain weave |
| 5 | Glass fabric 600 GSM | 600 GSM, 0.6 mm thickness,<br>Plain weave |
| 6 | Polyester Resin + Hardener (MEKP)+ Accelerator (Cobalt) | Mixing Ratio 100:1.5:0.5,<br>300 cp viscosity, RT curing |

### 4.1.2 Consumables and Accessories

VARTM process requires many types of consumables and accessories for the preparation of proper laminates. The consumable items are not integral part of laminate but they are part of the process, without which it is difficult to achieve the target result. The error in selection or not using correct consumables may result in improper impregnation. The following list shows consumables for VARTM set up.

**Braided transparent hose pipe**

- To supply vacuum from vacuum pump to resin trap and from resin trap to ball valve size 1/2 "

**Resin supply knob**

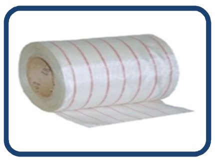
- To connect resin supply tube with spiral tube

**Connectors, T joint and Ball valves**

- To supply resin and vacuum, size 1/2 ", 3/8", 1/4"

**Resin stirrer sticks**

- To mix the resin with hardener



**Peel Ply**

- To remove components after curing from consumables
- Temperature range max up to 200 °, polyester, white colour

**High Permeable Distribution Media (HPM)**

- To increase flow velocity

**Vacuum Bag**

- For room temperature curing, transparent

**Resin mixing container**

- Size 1 Litre

The following list shows Accessories for VARTM set up. Accessories are required to enhance the performance of the process.

**Clamps**
- To hold hose pipe

**Scissor**
- Required to cut glass and carbon fabric and to trim laminates

**Electrical cutter**
- To cut glass and carbon fabric

**Portable hand grinding machine**
- 11000 RPM, 100 mm wheel size, 10 mm spindle size, 220 V

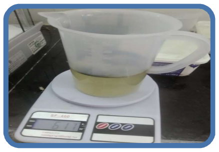
**Vacuum pump**
- Rotary vane type, oil lubricated, HP/220V,
- For vacuum $10^{-3}$ millibar required.

**Resin Trap**
- To ensure resin does not penetrate inside vacuum pump during impregnation. 29 cm ID and 30 cm OD, with vacuum gauge

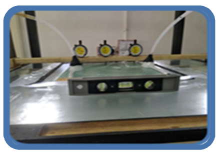
**Peristaltic pump**
- To supply resin at constant RPM

The following list shows measuring instruments used for VARTM set up.

**Weighing scale**
- Max capacity 220 g, least count 0.001 g

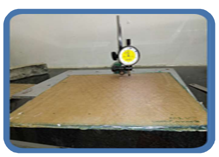


**Weighing scale**
- Maximum Capacity 5 kg, least count



**Spirit level**
- To measure flatness of table

**Fixture with three dial gauge**
- To measure thickness variation during the flow



**Dial gauge with magnetic stand and surface plate**
- To measure thickness variation in laminate

**Muffle furnace**
- Maximum temperature 1200 °C

**Crucible**
- For LOI and volume fraction measurement

**Density measuring kit**

## 4.2 CARE AND PRECAUTIONS DURING EXPERIMENTS

To get best quality laminates proper care and precautions should be taken while performing experiments. Figure 4.1 explains do’s and don’ts for using experimental facilitates.

[1]. Cover yourself with apron, mask, hand gloves, head cover and shoe cover while performing experiments.

[2]. Keep chemical in proper location and keep exotic chemical separate.

[3]. Keep exhaust fan on while using vacuum pump.

[4]. Keep the experiment room clean.

[5]. No food should be allowed inside laboratory.

[6]. Keep fire extinguisher in laboratory in case of emergency. And check expiry date regularly.

[7]. While cutting glass fabric use hand glows and cover your body fully.

[8]. Keep water supply as near as possible.


**Figure 4.1** Care and precautions during experiments

## 4.3 STEPS FOR VARTM PROCESS

Every process works on PDCA (plan, do, check and act) cycle. The experimental process steps can be divided in to three stages. The plan, do and check stages have been discussed below. The act stage is corrections that you take care of while performing next set of experiments.

### 4.3.1 Preparation stage

This is the first stage, also considered as "planning" stage, before starting actual laminate making process. Figure 4.2 demonstrates the various steps of readiness before starting actual impregnation.

1. Table cleaning with Acetone.
2. Application of masking tape.
3. Application of three coats of release mold spray.
4. Fabric cutting with template and making laminate number and laying direction.
5. Weighing of fabric layers.
6. Putting fabric layers on surface as per required orientation.
7. Application of Peel Ply layer.
8. Placement of High Permeable distribution Media.
9. Placement of spiral tube for resin supply and vacuum supply.
10. Vacuum bagging and sealing with sealant tape.

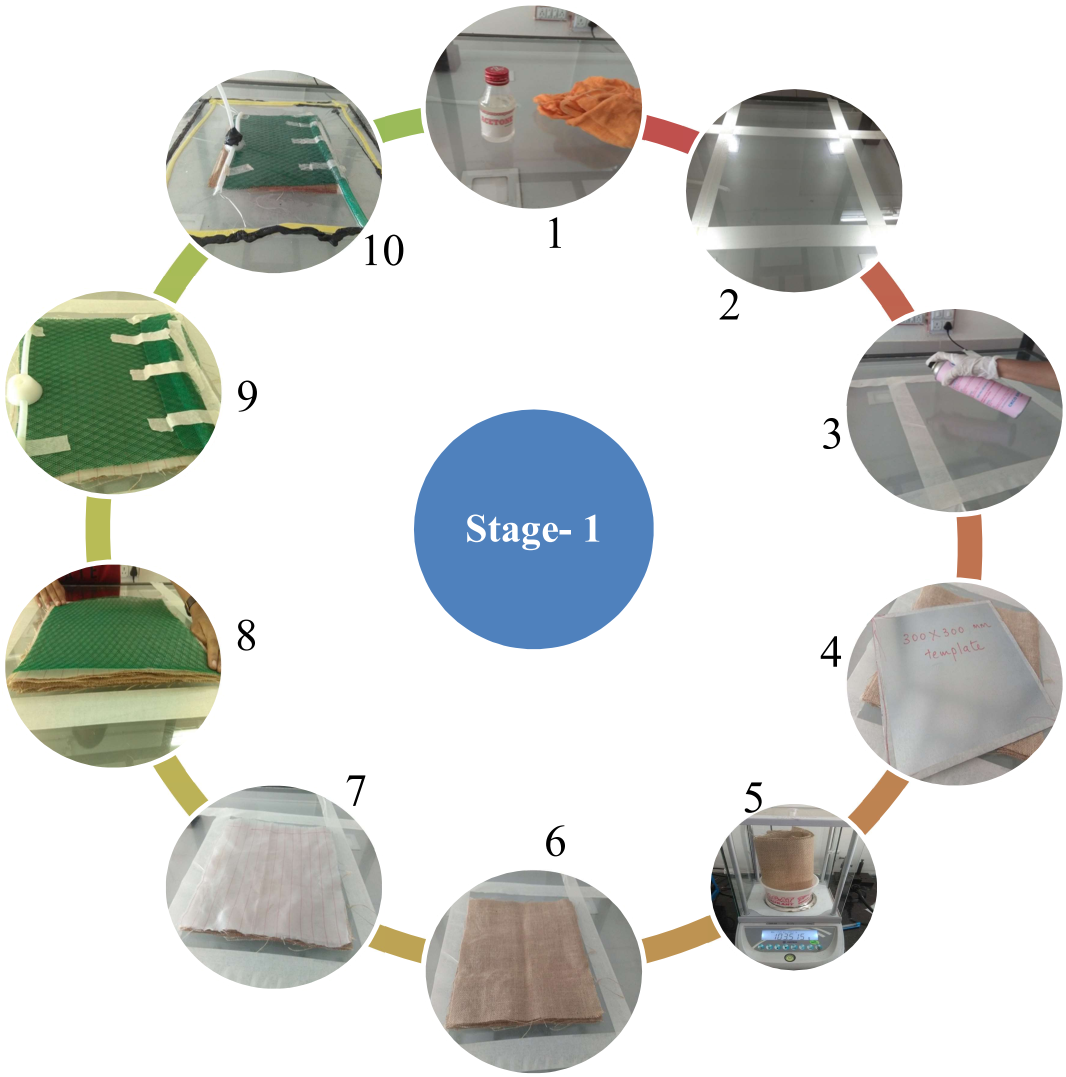



**Figure 4.2** Process steps - stage 1

### 4.3. 2 Actual process stage

After keeping setup ready the next stage is to perform actual experiment. This is the "do" stage. Figure 4.3 shows steps performed during this stage

1. Leak check
2. Resin weigh and resin stir
3. Resin impregnation
4. Pressure plate (Optional)
5. 24 hours Room temperature (RT) Curing

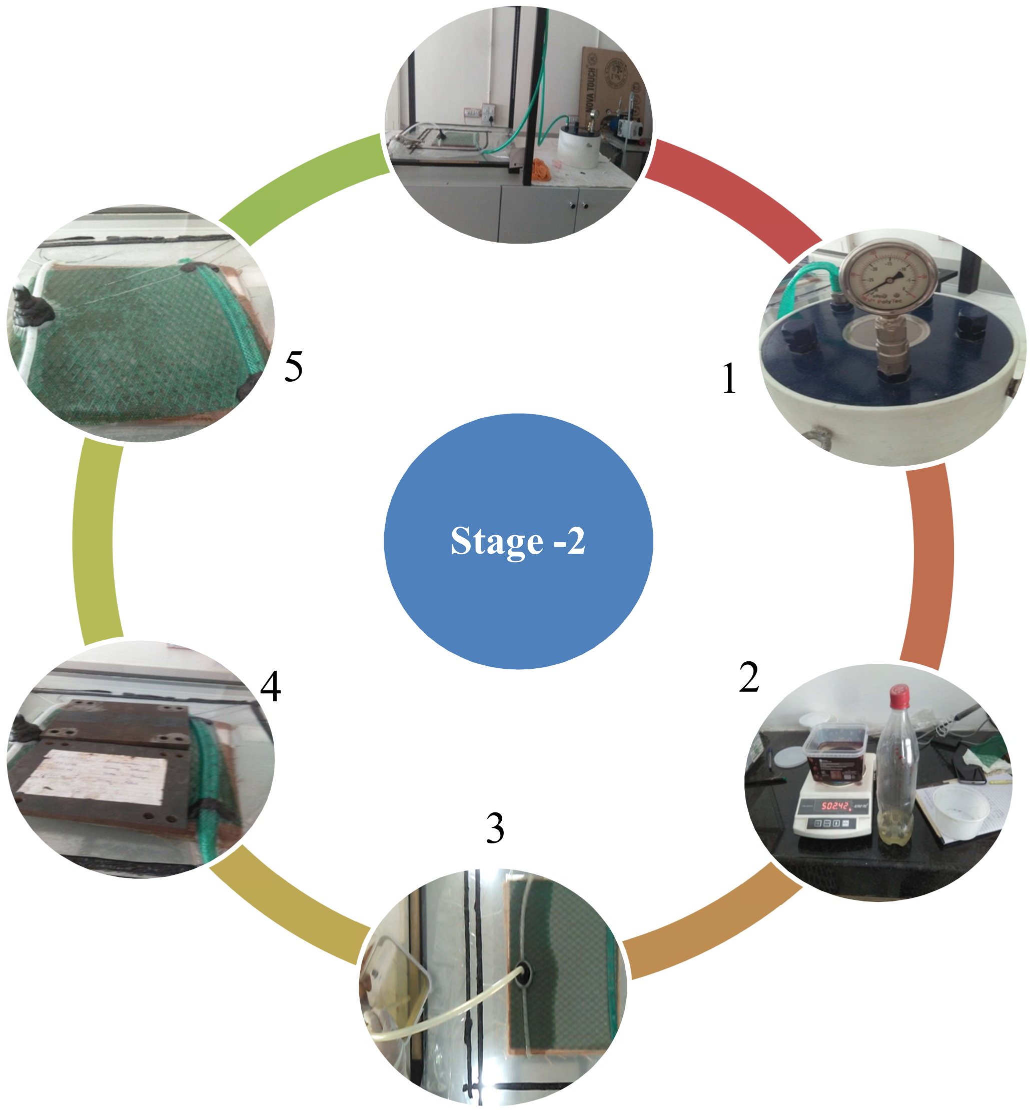



**Figure 4.3** Process steps - stage 2

### 4.3.3 Post process stage

The next stage is "check" stage. As shown in Figure 4.4, various destructive and non-destructive tests are performed.

1. Laminate extraction.
2. Laminate weighing.
3. Non Destructive Testing – Thickness check, Visual inspection
4. Destructive – Weight fraction, Volume fraction, Mechanical Characterization

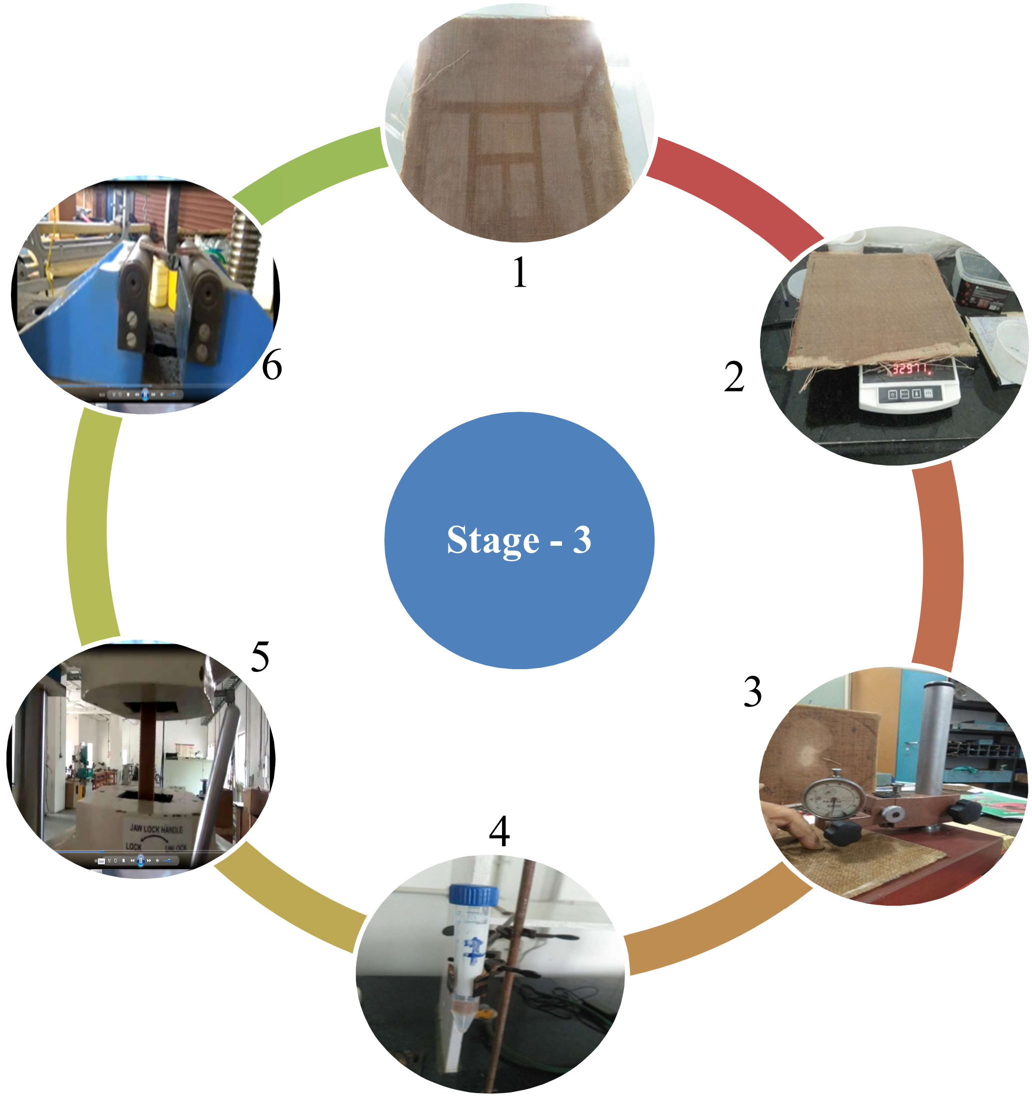



**Figure 4.4** Process steps - stage 3

## 4.4 DESIGN OF EXPERIMENTS

An experiment can be defined as the intentional change in input to observe variation in output of any system. By experiments we study the effect of parameters on an artificially controlled system (Mathews, 2005).

The design of experiment is the branch of applied statistics which deals with planning, conducting, analyzing and interpreting control tests to evaluate the factors that control the value of a parameter or a group of parameters. It is used to study effect of more than one variable and their interaction effect on system.

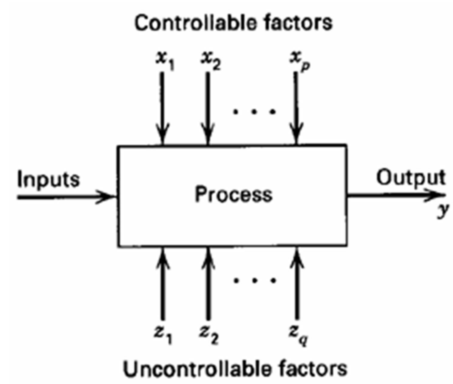



**Figure 4.5** Meaning of experiment. (Mathews, 2005)

The word design in design of experiments refers to the way in which variables are intentionally varied over many runs in an experiment. Once the experimental variables are identified and the levels of each variable are chosen, the experiment can be designed. Generally experiment design is expressed in the form of two matrices: 1) **Variable matrix**: Decides the variable and levels.2) **Design matrix**: Provide standard order run (gives the logical order of the experimental run) and Run Order column (The order of experimental run is always randomized).

The general steps in DOE includes preparation of cause and effect diagram, documenting process, defining problem statement, perform preliminary experiments, designing experiments by preparing parameter and design matrix, deciding sample size, blocking, and randomization, performing statistical analysis, interpreting result and performing final runs before submitting results.

There are various designs available to perform experiments. Full factorial and Taguchi Orthogonal array $L_9$ is used to design experiments for this thesis.

Factorial experiment allows the investigator to study the effect of each factor on the response variable, as well as the effects of interactions between factors on the response variable. If there are 3 factors 3 levels, the number of experiments for full factorial would be $3^3(I^k)=27$.

The mathematical model for these experiments will be

$$y = b0 + b1x1 + b2x2 + b3x3 + b12x12 + b13x13 + b23x23 + b123x1x2x3 + b11x1^2 + b22x2^2 + b33x3^2 \qquad (4.1)$$

**The Full Factorial Design** requires a large number of experiments to be carried out as stated above. It becomes laborious and complex, if the number of factors increase. To overcome this problem Taguchi suggested a specially designed method called the use of orthogonal array to study the entire parameter space with lesser number of experiments to be conducted. He proposed a set of experiments for given number of variables and their levels. In this thesis, $L_9$ Othorgnoal array has been used. Signal to noise ratio is used to anlyse the most significant variable and level of that variable. These experiments do not consider intraction effect and are mostly used as screening experiments.

Analysis of variance (ANOVA) is a collection of statistical models used to analyze the differences among group means and their associated procedures (such as "variation" among and between groups), developed by statistician and evolutionary biologist Ronald Fisher. It is used to compare simultaneously all possible pairs of means to see if there is difference between them.

To find the effect of one or more independent variables on dependent variable, Regression technique is used for modelling and analyzing. It generates mathematical equation for independent and dependent variables by showing the best line fit model. The experiment that involves quantitative predictors is usually analyzed by regression.

The coefficient of determination is a measure of how well the regression line represents the data. If the regression line passes exactly through every point on the scatter plot, it would be able to explain all of the variation. The coefficient of determination is the ratio of the explained variation to the total variation. The coefficient of determination represents the percentage of the data that is the closest to the line of best fit. For example, if $r = 0.922$, then $r^2 = 0.850$, which means that 85% of the total variation in y can be explained by the linear relationship between x and y.

In more complex regression problems where many independent variables and possibly interaction terms enter the model, it's unfair to measure the model quality with the coefficient of determination $r^2$. As more and more terms are carried in a complex model, the $r^2$ value will always increase. This makes it necessary to penalize $r^2$ for the additional complexity of the model. This new coefficient of determination, called the adjusted coefficient of determination, $r^2$ adjusted, is given as:

$$r^2_{adjusted} = 1 - \frac{df_{total} SS_{\varepsilon}}{df_{\varepsilon} SS_{y}} \qquad (4.2)$$

## 4.5 EXPERIMENTAL METHODOLOGY

The experiments are performed to observe the effect of input variables on output variables. The number of parameter affecting VARTM process has been identified from research review and total four set of experiments were performed along with preliminary experiments to study the effect of these variables in VARTM Process. While performing new sets of experiments, learning outcome of previous set of experiment was considered and some new modifications were applied in the experimental setup. Number of layers is selected as common variable parameter while performing experiments; however the layers selections are different for different set of experiments. The various set of experiments according to the order they were performed have been explained below.

Each experiment demonstrates material used and methodology followed during the experimentation. The experiments are performed either with hollow glass fabric (211 GSM), jute fabric (plain weave) or glass fabric (plain weave) as reinforcement and polyester resin (resin + hardener (MEKP) + accelerator (cobalt) in ratio of 100:1.5:0.5 as matrix.

### 4.5.1 Preliminary Experiment

Preliminary set up was developed to prepare laminate by VARTM process from hollow glass fabric (211 GSM) and polyester resin (Resin + Hardener (MEKP) + Accelerator (Cobalt)) in ratio 100:1.5:0.5. The consumables and accessories required for developing VARTM setup includes masking tape, mold release spray, peel ply, High Permeable distribution Media (HPM), vacuum bag, sealant tape, hose pipe, supply pipe and resin container, the accessories used is vacuum pump to supply vacuum, weighing machine to weigh the resin and scissor to cut the material.

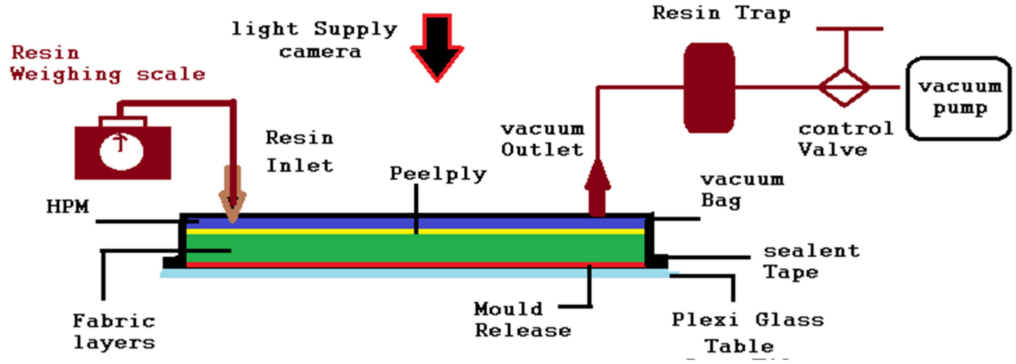



**Figure 4.6** Schematic diagram of VARTM preliminary experiment setup

Figure 4.6 shows the schematic diagram of VARTM preliminary experimental setup. Layers of fabric were laid on glass table after application of three coats of mold release spray. Peel ply was kept on layers of fabric and HPM was kept on peel ply. Inlet and outlet tubes were kept on HPM. The full assembly was covered with vacuum bag and sealed with sealant tape. After setting up, the vacuum supply was started and vacuum leak was checked. Resin was impregnated inside the fabric after weighing and mixing with hardener and accelerator. Resin supply was closed, once the resin reached the vacuum line. Vacuum pump was kept on until resin was cured. Figure 4.7 depicts picture of developed preliminary experimental setup.

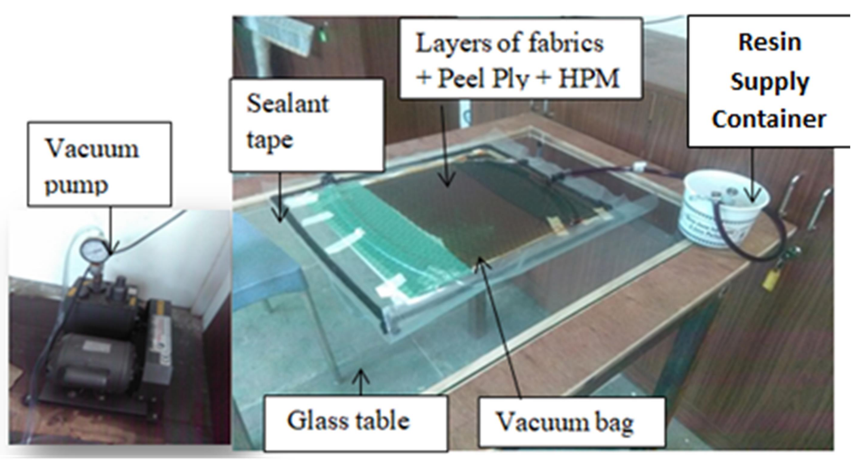



**Figure 4.7** Developed preliminary experimental setup

### 4.5.2 Investigations on Effect of Number of Layers, Position of Resin Supply and Location of Vacuum Supply for VARTM Process

Nine experiments were performed using experimental design, by Taguchi $L_9$ orthogonal array, for first set of experiments. The parameters selected were number of layers (4, 5 and 6), position of resin supply (top, middle and bottom) and location of vacuum supply (edge to centre, centre to edge and left to right). These parameters were chosen to understand effect of number of layers, pressure head during impregnation and flow direction on laminate quality.

Material used for this set of experiments was jute fabric (untreated, 290 GSM, 15X14 TPI) and polyester resin. The experimental set up is shown in Figure 4.8. The set up was modified by adding a frame, to keep a camera on top and light. Special frame was developed to put resin 500 mm top and 500 mm bottom, from table surface. Vacuum pump was directly connected to the experiment. Braded pipe was used to perform the experiment, so that vacuum could be sucked properly from the vacuum bag. The impregnation process was same as before.

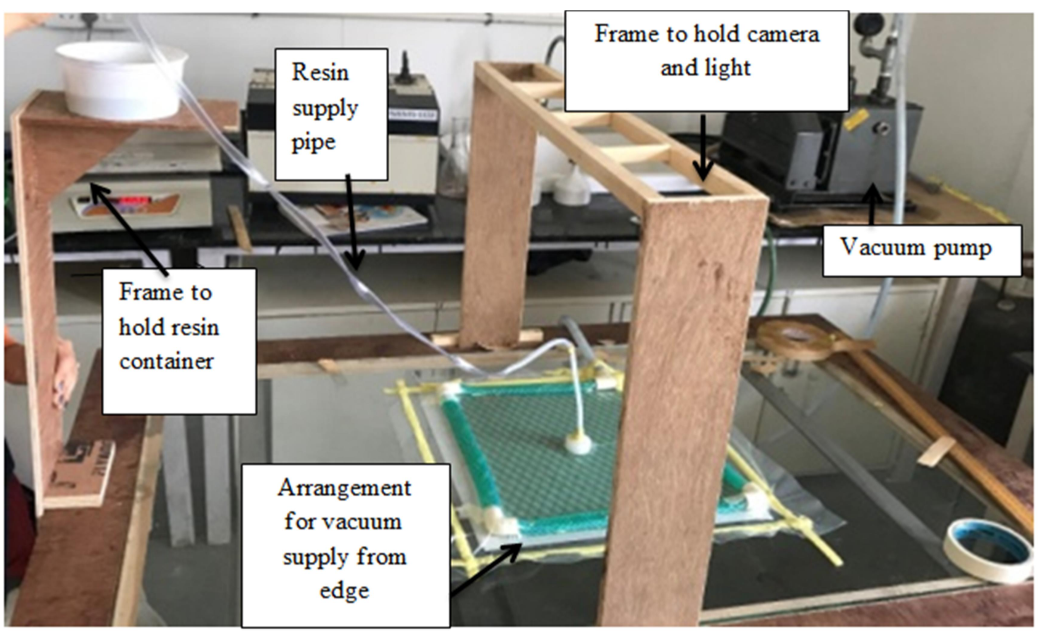



**Figure 4.8** Experimental setup

During performing these experiments, the concept of "Design of experiments" was learnt. Selection of tube was studied. Arrangement of vacuum line was studied and implemented. Fittings were used to connect the vacuum lines. Incomplete penetration and gelation before impregnation was observed initially in few laminates. Race tracking was also observed. Selection of testing method, ASTM code identification, test coupon marking and cutting, actual testing, consolidation of results and data analysis were part of learning.

For this experiment tensile, flexural, volume fraction and thickness variation was performed. It was understood that there is no standard method for finding volume fraction for natural fiber so solvent method was developed. THF (Tetra Hydro Furan) was selected as solvent for weight fraction measurement.

### 4.5.3 Preliminary Experiment on Developed Indigenous Experimental Setup

To incorporate study of maximum parameters and to realise its effect on various output conditions, the conceptualised drawing was prepared for VARTM experimental setup and the same was modelled in 3D as shown in Figure 4.9

The experimental setup includes impregnated area of one meter square. The extra space on two sides was provided, one for resin trap the other for resin supply container and peristaltic pump. The drawers are provided to store the materials and consumables. Proper lighting arrangements were made below the impregnation area for observing the resin flow. C - Frame was provided to fix camera on top to observe the resin flow. Slotting arrangements were made on the molding glass frame to affix level indicator. Support screws were provided to ensure proper levelling of mold surface during experiments. The molding surface was designed to tilt at desired angle with the help of frame supports and angle protractor arrangement. Refer Figure 4.10 to observe extra arrangements in the experimental setup.

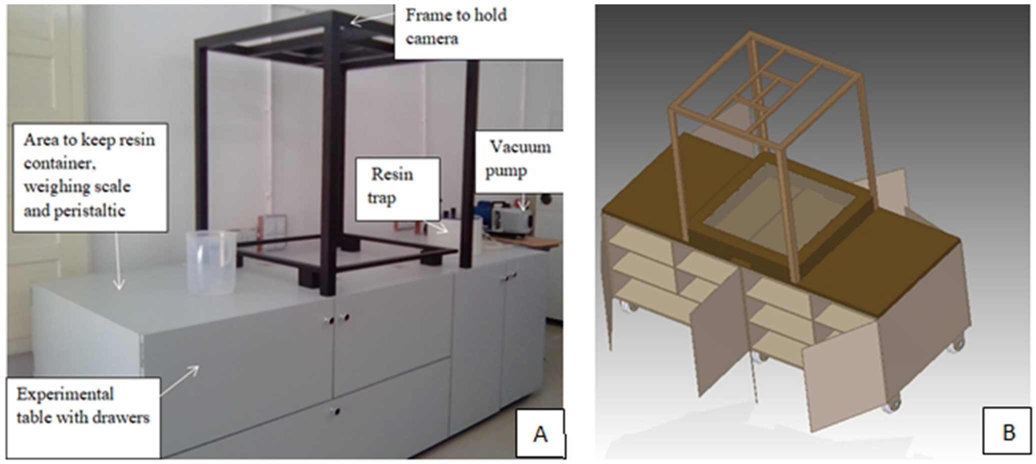



**Figure 4.9** a) 3D View of experimental setup b). Developed experimental setup

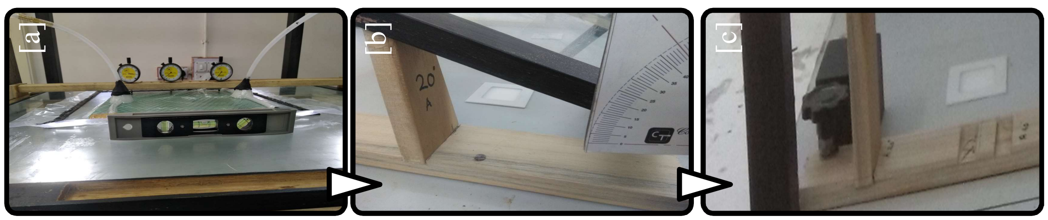



**Figure 4.10** a) slots for spirit level b) Arrangements for inclination c) Arrangement for levelling of table and lights

Preliminary experiment was performed with hollow glass fabric (211 GSM) and polyester resin to understand proper working and to determine short coming of the system. The preliminary experiment conducted on this indigenous set up is shown in Figure 4.11. The learning from the preliminary experiment has been incorporated in the next set of experiments. They are as follows:

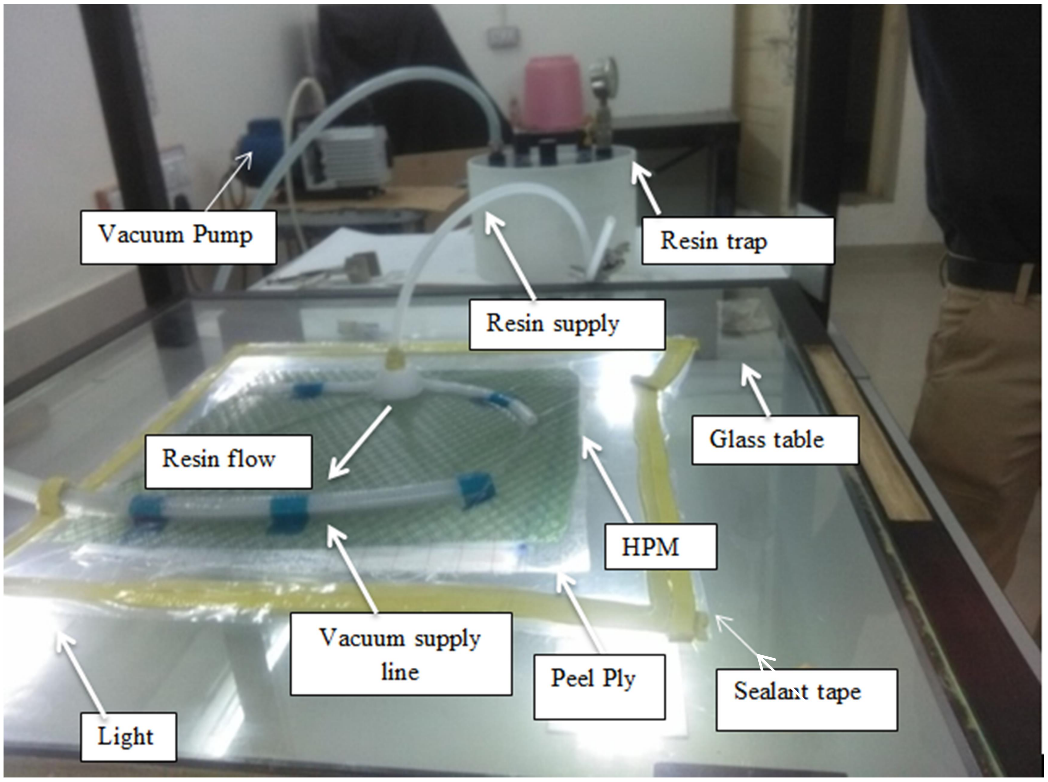



**Figure 4.11** Preliminary experiment conducted on indigenously developed setup

The resin trap should have container inside to collect the resin. The clamps to lock vacuum pipe and resin supply should be readily available. The supply of resin should be on the left side and vacuum supply should be on the right side. The length of vacuum pipe should be such that vacuum supply can be easily managed with the open-close valve. Proper care should be taken while sealing the vacuum bag. Camera and stopwatch should be readily available while performing the experiments.

### 4.5.4 Investigations on Effect of Number of Layers and Degassing for VARTM Process

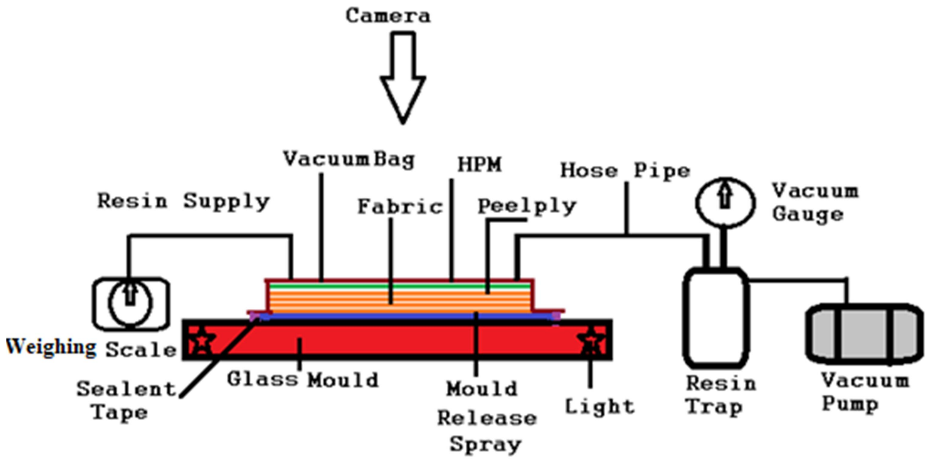



**Figure 4.12** Schematic diagram of effect of number of layers and degassing experimental set up

The schematic diagram of developed experimental setup for degassing is shown in Figure 4.12. Six set of experiments were performed three with degassing and three without degassing by changing number of layers from 5, 10 and 15. The laminates were prepared with jute fabric (untreated, 290 GSM, 15X14 TPI) and polyester resin. The resin was degassed for 3 minutes after mixing with hardener and accelerator. Degassing time was decided based on the amount of bubbles coming out from the resin. Degassing was done in resin trap at full vacuum. Tensile, flexural, 180x microscopic examination was performed on laminate; thickness variation was measured for cured laminate. Figure 4.13 depicts experimental setup for degassing. The impregnation process was same as before.


**Figure 4.13** Developed experimental setup with degassing

### 4.5.5 Investigations on Effect of Number of Layers, Inclination of Table and Amount of Vacuum Supply for VARTM Process

These experiments were performed with jute fabric (untreated, 290 GSM, 15X14 TPI) and polyester resin. Selected parameters and their levels were, number of layers (4, 8 and 12), amount of vacuum supply (29, 22 and 15 inHg) and inclination of table (0°, 20° and 40°). The schematic diagram of arrangements has been shown in Figure 4.14.

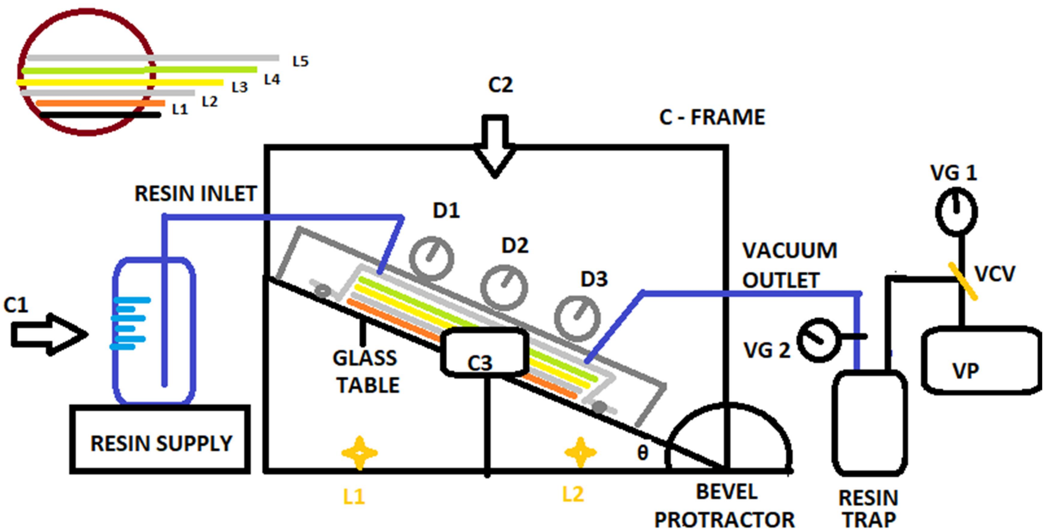



**Figure 4.14** Schematic diagram of effect of number of layers, inclination of table and amount of vacuum supply

Many modifications were included in the experiments. Special fixture was developed to observe thickness variation during flow which includes three dial gauges and specially designed stand to hold the dial gauge, on inclined plane. A fixture was developed to hold the glass frame at required angle (20° and 40°) along with bevel protractor. Two cameras were used to observe resin flow from top and from front. A control valve was introduced to control supply of vacuum during and after impregnation of resin. A scale was kept along the length of laminate to study the flow velocity from top and side camera. The impregnation process was same as before. The experimental setup is shown in Figure 4.15.

Tensile test, flexural test, thickness variation during flow, cured laminate thickness measurement, fiber weight fraction and flow velocity measurement was performed to study the effects of input parameters.

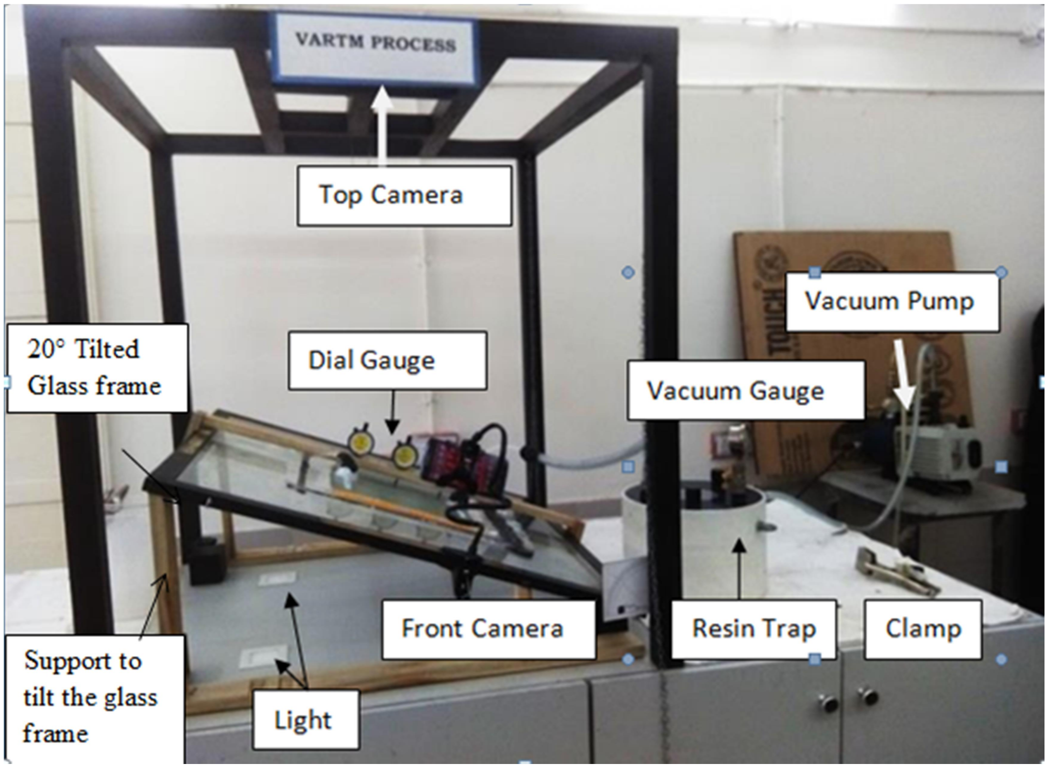



Figure 4.15 Experimental setup with variation in number of layers, inclination of table and amount of vacuum supply

### 4.5.6 Investigations on Effect of GSM of Glass Fabric, RPM of Peristaltic Pump and Amount of Vacuum Supply on VARTM Process

These set of experiments were done with the use of glass fabric (plain weave) and polyester resin. Full factorial approach was used to design experiments. Total 27 experiments were performed. The parameters selected were GSM of glass fabric (200, 400, and 600), RPM of peristaltic pump (70, 90 and 110) and amount of vacuum supply (15, 22 and 29 inHg). The Schematic diagram of arrangement is shown in Figure 4.16.

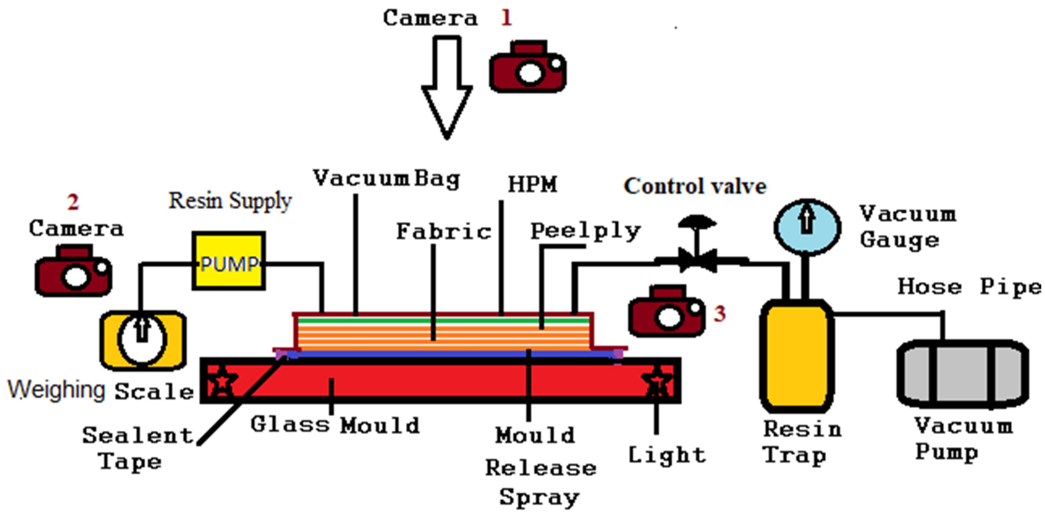



**Figure 4.16** Schematic diagram of experimental setup with peristaltic pump

Major modifications in these experiments were, use of peristaltic pump and glass fabric in place of jute fabric. Display check list was used to ensure nothing is missed during experiments. Scale and side camera were used to measure flow velocity during flow and dial gauge was used to measure thickness variation during flow. Weighing scale was used to measure flow rate during impregnation. Display of parameter near the layup was kept for record keeping. Spirit level was used to check the flatness of the table. Resin was degassed before impregnation. 15 minutes pre-compaction of fabric was performed. Control valve was used between resin supply tube and hose pipe which was connected to resin trap.

Tensile, flexural and fiber volume fraction measurement with LOI method were performed. The tensile test coupons were provided with tabs before performing tensile test. Flow rate, flow velocity and thickness variation during and after curing of laminate was also performed. Figure 4.17 explains the experimental setup arrangement. The impregnation process was same as before.

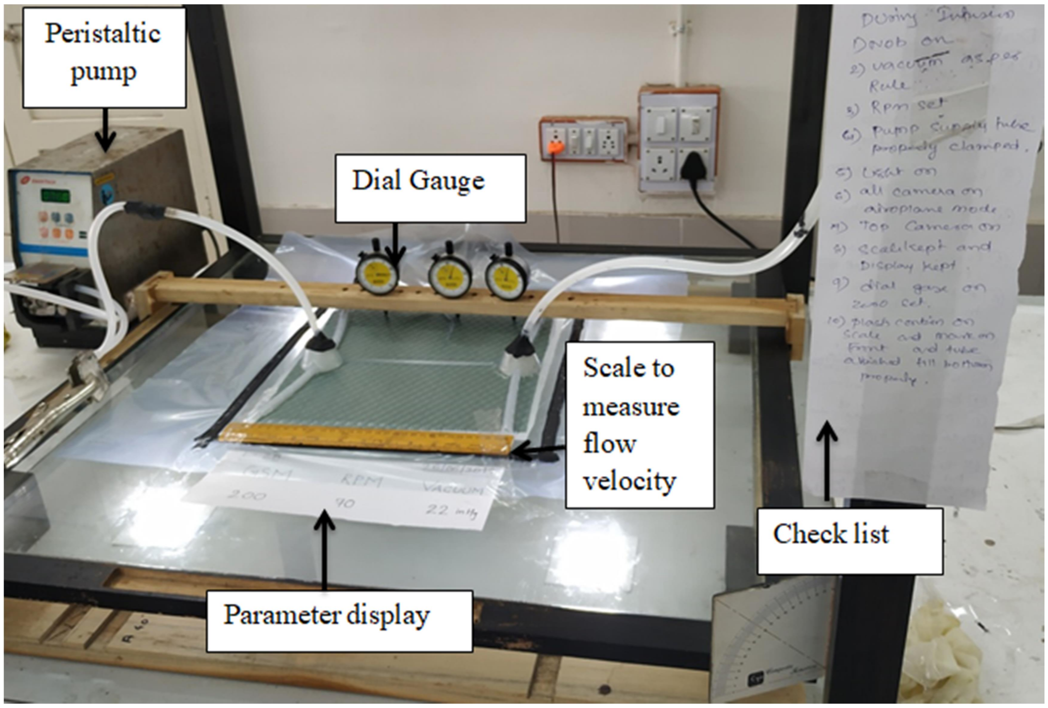



**Figure 4.17** Experiment set up with variation in GSM, RPM of peristaltic pump and amount of vacuum supply