CHARACTERISATION OF NANOPARTICLE THROUGH SEM, FTIR, XRD & DSC

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Abstract
Nanoparticles are widely due as Sustained Release Drug Delivery System. Due to their smaller size of controlled drug release potential, targeting ability, enhancement of therapeutic efficacy and reduction of toxicity. Nifedipine one of the calcium channel blocker use for the management of hypertension. Spray drier technique develops Nifedipine loaded nanoparticle. Primary superimulsion phase contains Drug (Nifedipine), Polymer (Ethylcellulose) & suitable non-aqueous Solvent (Dichloromethane) which are sprayed over secondary phase containing aqueous solvent with dispersing polymers (PVA) are prepared by taking drug to polymer weight ratio of 20:90. Formation of films on the surface of secondary phase dried, sieved & forward to spectroscopic analysis. SEM micrograph shows the morphology of particles. FTIR spectrometer as an analytical tool shows that the final formulation retains its chemical groups with nucleus (dihydropyridine) indicates no fluctuation at drug stability. DSC study shows the glass transition remains likely constant. X-Ray study shows the formulation looses its crystallinity.

Key Wards: Nanoparticles, Drug Delivery, Nifedipine, Spray drier technique

Introduction
Drug delivery system (DDS) concept is not new in the treatment of various diseases. Targeting delivery of drugs to convey a sufficient dose of drug to the lesion, suitable carriers of drugs were needed. These nano & microparticle carriers have important potential applications for the administration of therapeutic molecule [1]. Nifedipine (Nif.) Is a prototype Dihydropyridine calcium channel blocker with a rapid unset and short duration of action. Inhibiting passage of calcium through the voltage gated L-type (for Large/Long-lasting current) calcium channel on vascular smooth muscle cells and cardiac myocytes, reducing calcium availability for muscle contraction [2]. Nanoparticles may be exhaled as a result of their small size; however, they are desired to enhance the dissolution rate of poorly soluble drugs[3]. These possible benefits include controlled release, protection of the active pharmaceutical ingredient and drug targeting. Nanoparticles are expected to offer new solutions e.g. for gene therapy and delivery of peptide drugs [4]. Spray-drying is a well-known technique to produce powders, granules or agglomerates from the mixture of drug and excipient solutions as well as suspensions. Various process parameters are to be controlled to get the desired size of particles.
Particle size depends upon the size of nozzle, spray flow rate, atomization pressure, inlet air temperature and extent of cross linking [5].

**Material & Method**

**Chemicals required:** Nifeedipine (J.B Pharma [USP]), ethyl cellulose (Merch [USP]), polyvinyl alcohol (S.D. fine [IP]), Acetone (Merch).

**Preparation:** Nifedipine and ethylcellulose with total with 0.5 g were brought to make an polymeric suspension in 30 ml acetone in as primary phase in the ratio of 20:90. The suspension was sprayed over the fluid bed containing solution of 0. 3 % w/v of polyvinyl alcohol acts as secondary phase. These sprayed particles were spread over the bed where the diluents evaporate rapidly at room temperature. The mass was filtered from bed & dried. Formation of particles were separated through 120 # sieve and collected for routine analysis.

![Figure 1. Nanoparticles of irregular size](image)

**Result & Discussion**

**Differential scanning calorimetry (DSC)**

Differential scanning calorimeter (DSC) analysis was carried out using a thermal analysis system (Mettler DSC 822 model). Calibration with the standard (indium) was undertaken prior to subjecting the samples for study (between 35-400 °C) with sample purge flow 40 ml/min, which were heated at 10o C/min in an aluminum pan under a nitrogen atmosphere while using an empty pan as the reference in this instrument. The instrument automatically calculated onsets of melting point and enthalpy of fusion.

**Table 1 Thermal data of Nifedipine Nanoparticles**

<table>
<thead>
<tr>
<th>Sample/Properties</th>
<th>Nifedipine Particles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity</td>
<td>13.600 mg</td>
</tr>
<tr>
<td>Heating rate</td>
<td>10 °C /min</td>
</tr>
<tr>
<td>GLASS TRANSITION</td>
<td></td>
</tr>
<tr>
<td>Onset</td>
<td>166.37 °C</td>
</tr>
</tbody>
</table>
**FTIR Spectroscopic Analysis**

The infrared spectra are recorded on Fourier Transform Spectrometer in the mid–infrared region (MIR) within the range (400-4500 cm\(^{-1}\)). Due to the complex interaction of atoms within the molecule, IR absorption of the functional groups may vary over a wide range. However, it has been found that many functional groups give characteristic IR absorption at specific narrow frequency range. Multiple functional groups may absorb at one particular frequency range but a functional group often gives rise to several characteristic absorptions. Stretching & bending vibrations are varied after formulation can be observed. Thus, the spectral interpretations should not be confined to one or two bands only actually the whole spectrum should be examined [6, 7].

![FTIR of nifedipine nanoparticles](image)

The FTIR band at 3415 cm\(^{-1}\) represented O-H group stretching of O-H, H-bonded single bridge. In the region of 3021 cm\(^{-1}\) is due to stretching vibrations of Ar-H, (-CH) several band at 2911 cm\(^{-1}\) (C-H), 2 or 3 band of methyl group. The presence of aryl carboxylic group in the region 1603 cm\(^{-1}\) represents C=O stretching vibration. 1617 cm\(^{-1}\) represents presence of pyridine nucleus ring breathing. Presence of aryl nitro group in the region of 1534- 1467 cm\(^{-1}\) is also observed. Etherial group is found at 1115 cm\(^{-1}\), where C-O-C shows very strong stretching.

**X-Ray Analysis**

XRD pattern is characterized by the interplanar d- spacing and the relative intensities (I/I_0) of the strongest peaks in the pattern under the Hanawalt system. It was found out the position of values of product crystallinity or amorphic nature. Data helps in finding out the fingerprinting region of relative intensity with respect to d-spacing values.
The broad peak shows that the formulation of Nifedipine Nanoparticles partially crystalline in nature. The broad peaks observed in the diffractogram at around 20.2° for Nifedipine in the formulation. The crystallinity decreases during preparation.

**Conclusion**
From the above discussion it was concluded that the easy method of spray drying technique used to prepare nanoparticle which can be loaded drug as better carrier. The analytical techniques show the figure print characteristic value about thermal & spectroscopic mean. The FTIR states the stability of groups of drug molecule present after preparation will shows proper therapeutic effect.

**References**


