Lallemantia reylenne seeds as superdisintegrant: Formulation and evaluation of nimesulide orodispersible tablets

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Abstract

Aim: Orodispersible tablets also known as fast dissolving tablets disintegrate instantaneously within the mouth and thus can be consumed without water. The present study was aimed to formulate orodispersible tablets of nimesulide by using Lallemantia reylenne seeds as natural superdisintegrant. Materials and Methods: Powdered lallemantia seeds were characterized for powder flow properties (bulk density, tapped density, carr’s consolidation index, hausner ratio, angle of repose), swelling index, viscosity, pH, and loss on drying. The prepared tablets were evaluated for different tablet parametric tests, wetting time, water absorption ratio, effective pore radius, porosity, packing fraction, in vitro and in vivo disintegration time, in vitro dissolution and stability studies. Results and Discussion: Increase in Lallemantia reylenne concentration had an appreciable effect on tablet hardness and friability which clearly indicated binding potential of the seeds. Water absorption ratio increased with increase in Lallemantia reylenne concentration from batch A1 to A4. Water uptake coupled natural polymer swelling could be the most probable mechanism for concentration dependent reduction in disintegration time by the Lallemantia reylenne seeds. Porosity of the formulated tablets was found to increase from batch A1-A4. The in vitro disintegration results were in line with in vivo disintegration results. Conclusion: It could be concluded that Lallemantia reylenne seeds could be used as natural superdisintegrant in the formulation of orodispersible tablets.

Key words: Fast disintegrating tablets, Lallemantia reylenne, superdisintegrant

INTRODUCTION

Plant products nowadays are widely used as an alternative to synthetic products due to ease of local accessibility, lower prices as compared to synthetic products, biocompatible, biodegradable nature, and environment friendly nature. The oral route of drug delivery is the most preferred route of administration of drugs for systemic action. The tablet is the most widely used oral dosage form because of its convenience in terms of self-administration, compactness, non-invasive, and economical to manufacture. About one third of the population including paediatrics, bed ridden patients, and geriatrics face swallowing problems related to solid oral dosage forms resulting in poor compliance to drug therapy. Novel drug delivery systems are aimed to produce effective drug therapy with better patient compliance, efficacy, and safety. Fast disintegrating drug delivery systems offer a solution to overcome swallowing problems of these patients. Orodispersible tablets are commercially prepared by direct compression technology and makes use of superdisintegrants, which when placed on the tongue disintegrates instantaneously and the drug gets dissolved or dispersed in saliva.[1] The technologies used for preparation of orodispersible tablets include lyophilization,[2] moulding,[3] direct compression,[4] cotton candy process,[5] spray drying,[6] sublimation,[7] and nanonization.[8] These techniques are based on the principles of increasing porosity and/or addition of superdisintegrants and water soluble excipients in the tablets, which aid in disintegration of tablets within seconds.[9] Synthetic superdisintegrants such as sodium starch glycolate, cross carmellose sodium, and crosspovidone have swelling and capillary based action, which aids in quick disintegration of the tablet. Another mechanism of super disintegration of tablets

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Characterization of *Lallementia Reylenne* seeds

**Evaluation of powder flow properties**

The *Lallementia reylenne* powdered seeds were evaluated for flow properties including bulk density, tapped density, angle of repose, Carr’s compressibility index, and Hausner ratio.

**Swelling index**

The study was carried out using a 100 ml stoppered graduated cylinder. The initial bulk volume of 1 gm powder seeds was noted. Water was added in sufficient quantity to produce 100 ml of a uniform dispersion. The sediment volume of the swollen mass was measured after 24 hour, stored at room temperature. The swelling index was calculated as

\[
\text{Swelling index} = \frac{V_2 - V_1}{V_1} \times 100
\]

where, \(V_1\) and \(V_2\) are initial volume of material before hydration and volume of hydrated material, respectively.

**Viscosity**

Viscosity of 1% solution of (w/v) *Lallementia reylenne* seeds was measured at 37 ± 1°C using a searle type viscometer, DV-2 + LV Brookfield Viscometer, USA with spindle number 62 at different rpm.

**Determination of pH**

The pH of 1% solution of (w/v) powdered *Lallementia reylenne* seeds was determined using digital pH meter (EI products, India) at 37°C.

**Loss on drying**

Loss on drying technique (LOD) is used to determine high levels of moisture or solvents present in the sample. The material sample was weighed (\(W_1\)) and heated in an oven for 2 hrs. Sample was cooled in the dry atmosphere of a desiccator, and then reweighed (\(W_2\)). % LOD was calculated by

\[
\% \text{ LOD} = \left( \frac{W_1 - W_2}{W_1} \right) \times 100
\]

**Preparation of tablets**

Orodispersible tablets containing 100 mg of nimesulide were prepared by direct compression method and the different formulae employed in the study are shown in Table 1. The drug and excipients were passed through 60 mesh sieve to ensure better mixing. Avicel PH 101 was used as a directly compressible diluent. The directly compressible mixture were compressed using multipunch tableting machine (AK Industries, India) fitted with 8.40 mm flat faced punch and die set possessing 50 ton compression force. Before compression, the surface of die and punch were lubricated with magnesium stearate. About 300 tablets were made for each batch.

**Evaluation of tablets**

**Diameter and thickness**

A calibrated vernier calliper (Indian calliper industries, Ambala, India) was used to evaluate diameter and thickness of tablets.

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**Materials and Methods**

**Materials**

Nimesulide and croscarmellose sodium were received as gift samples from Park Pharmaceuticals, Baddi, India. Avicel PH-101 was procured from Sigma Aldrich, USA. Talc and magnesium stearate were purchased from S. D. Fine Chemicals Ltd. Mumbai, India. *Lallementia reylenne* seeds were purchased from Yarrow Chem, Mumbai, India. *Lallementia reylenne* seeds were powdered and passed through 60-mesh sieve for being used in the research work. All other chemicals and reagents were of analytical grade and were used as such.
Hardness
The hardness of the tablets was determined by using Monsanto hardness tester (Pharma Chem Machineries, Mumbai, India). A tablet hardness of about 4–5 kg/cm² is considered adequate for mechanical stability.

Friability
As per USP 30-NF 25, twenty six tablets were taken, which corresponded to 6.5 g weight. They were placed in a Roche friabilator and were rotated at 25 rpm for 4 minutes. The tablets were taken out, dedusted, and reweighed. The percentage friability of the tablets was calculated by the formula,

\[
\text{Percentage friability} = \frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \times 100
\]

Measurement of tablet tensile strength
The tablet tensile strength is the force required to break a tablet by compressing it in the radial direction and is measured using a monsanto hardness tester. For measuring the hardness of the tablets, the plunger of the hardness tester is driven down at a speed of 20 mm/min. Tensile strength for crushing (T) is calculated using equation:

\[
T = \frac{2F}{\pi dt}
\]

Where F is the crushing load, and d and t signify the diameter and thickness of the tablet, respectively.

Weight variation test
A total of 20 tablets from each batch were subjected to weight variation test. As per Indian Pharmacopoeia standards, the tablets should be within the specified limits, i.e., ± 5% of average weight.

Wetting time
A piece of tissue paper (10.75×12 mm) folded twice was placed in a culture dish (d = 6.5 cm) containing 6 ml of simulated saliva (phosphate buffer pH 6.8). A tablet was carefully placed on the surface of tissue paper and the time required for simulated saliva to reach the upper surface of the tablet was noted as the wetting time.[19]

Water absorption ratio
Test was done with the same procedure as that of wetting time. In this test, initial weight of tablet was noted before placing on petri dish. After complete wetting the wetted tablet was then weighed. Water absorption ratio, R was determined using the equation,

\[
R = \frac{100 \left( W_t - W_a \right)}{W_b}
\]

Where, \( W_a \) is weight of tablet after water absorption and \( W_b \) is weight of tablet before absorption.[19]

Effective pore radius
Effective pore radius (\( R_{\text{eff.P}} \)) of the powder blend was determined using method reported by Rana et al.[20] In short a micropipette tip (2 ml, transparent) was completely filled with powder and weighed (\( W_t \)). Then n-hexane (surface tension (\( \gamma \)) 18.4 mN/m) was poured dropwise on bedtop till the solvent filtered out at the bottom of the tip. The tip was reweighed (\( W_f \)). The experiments were repeated 3 times.

\[
R_{\text{eff.P}} = \frac{W_t - W_f}{2\pi \gamma}
\]

Porosity
Porosity is a measure of the void spaces in a material, and is a fraction of the volume of voids over the total volume. Its value ranges between 0–1, or as a percentage between 0–100%.

The porosity of the tablets was calculated as follows:

\[
\varepsilon = 1 - \frac{m}{P^*_{\text{true}} V}
\]

where \( P^*_{\text{true}} \) is the true density of the mixture, m and V are the weight and volume of the tablet, respectively. The true density of the powder was determined using true density meter (SMART PYCNO 30). When helium gas is used, initially vacuum is necessary to remove air from the pores of the sample. After that purging with helium gas is done. Then the normal procedure is followed. Two pressure readings were used to calculate true density. Initially helium gas was pressurised in a known reference volume. This reading was taken as first pressure reading. Then the gas was allowed to pass to a sample cell containing the sample material, which resulted in pressure drop as compared to initial pressure and this dropped pressure was taken as second pressure reading. Then material volume was calculated from which true density was calculated.

Table 1: Composition of Nimesulide orodispersible tablet formulations

| Ingredients (mg) | A1 | A2 | A3 | A4 | B1 | B2 | B3 | B4 |
|------------------|----|----|----|----|----|----|----|----|
| Nimesulide       | 100| 100| 100| 100| 100| 100| 100| 100|
| Lallemantia reylenne powder | 6.25| 12.5| 18.75| 25| -| -| -| -|
| Croscarmellose sodium | -| -| -| -| 6.25| 12.5| 18.75| 25|
| Avicel PH 101    | 138.75| 132.5| 126.25| 120| 138.75| 132.5| 126.25| 120|
| Talc             | 2.5| 2.5| 2.5| 2.5| 2.5| 2.5| 2.5| 2.5|
| Magnesium stearate | 2.5| 2.5| 2.5| 2.5| 2.5| 2.5| 2.5| 2.5|
| Total weight     | 250| 250| 250| 250| 250| 250| 250| 250|
gravity (SG) bottle filled with liquid paraffin and the weight of the SG bottle containing a sample of the drug powder (1 g) was noted. Then the SG bottle was filled with liquid paraffin. The final weight was determined. The determination was performed in triplicate, and mean results were used in the calculation of Pf. If the packing fraction is very high, fluid is unable to penetrate in the tablet, which leads to slower disintegration.\cite{21}

In vitro disintegration time

Disintegration time for orodispersible tablets was determined using USP disintegration apparatus with simulated saliva as the disintegrating medium (Phosphate buffer pH 6.8, 900 ml at 37°C) at a constant frequency of between 28 and 32 cycles per minute through a distance of 50 to 60 mm. To comply the test, all tablets should disintegrate within 3 minutes as per official requirements.

In vivo disintegration time

In vivo disintegration time\cite{20} was judged on five healthy male volunteers, for each batch of orodispersible tablets. The purpose of study was previously informed to the. Prior to the test the volunteers were instructed to rinse their oral cavity with distilled water. Each volunteer was asked to place one tablet on the tongue and stopwatch was started immediately. Volunteers were strictly told not to chew or swallow the tablets, licking was allowed. The test was concluded when there were no lumps left in the oral cavity, after which they were told to rinse there mouth properly.

Moisture uptake studies

Due to presence of hydrophilic excipients, orodispersible tablets are prone to attract moisture from the environment. This calls for need of special attention towards their storage and packaging. Therefore, moisture uptake studies are strongly recommended for orodispersible tablets.\cite{21} The test was performed by keeping ten tablets in a desiccator (containing calcium chloride) for 24 hours at 37°C to assure complete drying. The tablets were then weighed and stored for 2 weeks at 75% humidity. To achieve required humidity, a saturated solution of sodium chloride was kept at the bottom of the desiccator for three days. On the tenth day, tablets were re-weighed and the percentage increase in the weight was recorded.

Drug content

Twenty tablets were weighed and powdered. An amount of the powder equivalent to 100 mg of nimesulide was dissolved in 100 ml of pH 8.4 alkaline borate buffer, filtered, diluted appropriately, and analysed for drug content at 397 nm using UV-Visible spectrophotometer (Systronics 2202, India).\cite{23}

In vitro dissolution studies

For in vitro drug release studies, an eight stage USP dissolution apparatus II (Lab India, DS 8000) was used. The dissolution test was performed using 900 ml of alkaline borate buffer (pH 8.4) at 37 ± 0.5°C. The speed of rotation of paddle was set at 100 rpm. At a predetermined time interval, 5 ml samples were withdrawn, filtered through Whatman filter paper, adequately diluted, and analyzed using UV-Visible spectrophotometer (Systronics 2202) at 397 nm. All experiments were run in triplicate.

Stability testing

The prepared batches were evaluated for stability studies. During the full duration of study temperature and relative humidity of about 40 ± 2°C and 75% RH, respectively were maintained. The formulations were analysed at 0 day, 1 and 3 month time interval for hardness, friability, tensile strength, drug content, and in vitro disintegration time.

RESULTS AND DISCUSSION

Characterization of Lallemantia reylenne seeds

Powder flow properties, swelling index, viscosity, pH, and loss on drying (LOD) were studied for characterizing the powdered Lallemantia reylenne seeds. The results of powder flow properties [Table 2] clearly indicate good flow characteristics. Swelling index was found to be 1400, which point towards good swelling capability of Lallemantia reylenne seeds. Viscosity of 1% w/v solution at 37 ± 1°C of seeds of Lallemantia reylenne, using spindle number 62 of Brookfield viscometer was found to be 60, 90, 120, 150, 235, and 302 centipoise at 1.5, 3, 6, 12, 30, and 60 rpm, respectively. Plots between shear stress and rate of shear were plotted which clearly indicates obedience to newtonian flow [Figure 1]. Powdered Lallemantia reylenne solution (1% w/v) at 37 ± 1°C exhibited a pH of 6.42. Loss on drying was obtained to be 10.85%. Owing to its good swelling and powder flow properties, Lallemantia reylenne seeds were further evaluated as a superdisintegrant in nimesulide orodispersible tablets.

Evaluation of tablets

All the batches of orodispersible tablets were formulated under similar conditions to avoid processing variables. The tablets prepared by direct compression method were found to be free from capping, chipping, and sticking. The prepared tablets were evaluated for various physical parametric tests. The diameter and thickness [Table 3] of tablets was found to be 8.39 ± 0.05 to 8.41 ± 0.04 mm (A1-A4), 8.42 ± 0.04 mm to 8.41 ± 0.03 mm (B1-B4), and 4.71 ± 0.03 mm to 4.78 ± 0.04 mm (A1-A4), 4.81 ± 0.06 mm to 4.80 ± 0.05 mm (B1-B4), respectively. An appreciable effect was seen on tablet hardness, friability and tensile strength due to increasing concentration of Lallemantia reylenne. Hardness and friability [Figure 2] were found to be 2.84 ± 0.66 to 4.80 ±

| Parameters | Results |
|------------|---------|
| Bulk density (g/cm³) | 0.46 |
| Tapped density (g/cm³) | 0.55 |
| Carr’s compressibility index (%) | 16.36 |
| Angle of repose | 26.56 |
| Hausner ratio | 1.21 |
| Swelling index | 1400 |
| pH | 6.42 |
| LOD (%) | 10.85 |
0.35 kg/cm² and 0.59 ± 0.04 to 0.24 ± 0.03%, respectively, clearly indicating binding potential of Lallemantia reylenne seeds. Tensile strength (parameter of mechanical integrity of tablets) was found to increase from 0.457 ± 0.08 to 0.760 ± 0.10 MN/m², respectively, confirming binding capability of Lallemantia reylenne seeds. All of the batches passed weight variation test.

Wetting time, water absorption ratio, and in vitro disintegration time were found to be ranging between 18 ± 2 to 10 ± 3 seconds, 59.11 ± 0.65 to 85.76 ± 0.96, and 20 ± 1 to 12 ± 1 seconds, respectively. Water absorption capacity was found to increase with the increase in concentration of Lallemantia reylenne seeds from batches A1 to A4, which could be due to higher water uptake by the natural polymer. Water uptake coupled natural polymer swelling could be the most probable mechanism for concentration dependent reduction in disintegration time by the Lallemantia reylenne seeds. Hence, the formation of highly wettable/porous structure upon contact with water may be the best possible reason for fast disintegration achieved by inclusion of powdered Lallemantia reylenne seeds in the orodispersible tablets.

Reff.P is an indicator of tablet porosity. Reff.P and porosity [Figures 3 and 4] were found to be ranging from 3.122 ± 0.45 to 4.126 ± 0.31 mm (A1 to A4) and 15.522 to 24.576%, respectively, indicating appreciable capability of Lallemantia reylenne seeds to increase water penetration due to wicking action, which increases porosity thus lowers disintegration time with increase in polymer concentration. So these tests clearly depicted the potential of Lallemantia reylenne seeds to increase the porosity of tablets with increase in its concentration. Tablet packing fraction was found to be 0.822, 0.805, 0.786, and 0.754 (A1–A4), which indicates towards tablet superdisintegrant property of Lallemantia reylenne seeds. Figure 5 depicts the disintegration pattern of orodispersible tablets formulated using Lallemantia reylenne seeds as superdisintegrant. Five healthy male volunteers were selected to judge in vivo disintegration time. The in vivo disintegration time was found to be 22 ± 2 to 11 ± 2 seconds (A1–A4). In vivo performance of the formulated orodispersible tablets using Lallemantia reylenne as superdisintegrant was well in line with the in vitro results.

### Table 3: Evaluation of the prepared tablets

| Parameters                  | A1    | A2    | A3    | A4    | B1    | B2    | B3    | B4    |
|-----------------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| Diameter (mm)               | 8.39±0.05 | 8.42±0.03 | 8.44±0.02 | 8.41±0.04 | 8.42±0.04 | 8.45±0.02 | 8.42±0.05 | 8.41±0.03 |
| Thickness (mm)              | 4.71±0.03 | 4.75±0.04 | 4.73±0.02 | 4.78±0.04 | 4.81±0.06 | 4.79±0.07 | 4.80±0.02 | 4.80±0.05 |
| Friability (%)              | 0.59±0.04 | 0.48±0.05 | 0.38±0.01 | 0.24±0.03 | 0.84±0.06 | 0.87±0.04 | 0.70±0.02 | 0.68±0.01 |
| Hardness (kg/cm²)           | 2.84±0.66 | 3.40±0.27 | 4.10±0.82 | 4.80±0.35 | 2.56±0.72 | 2.87±0.49 | 3.01±0.77 | 3.40±0.56 |
| Tensile strength (MN/m²)    | 0.457±0.08 | 0.541±0.12 | 0.654±0.05 | 0.760±0.10 | 0.402±0.09 | 0.454±0.07 | 0.475±0.14 | 0.537±0.11 |
| Weight variation test       | Pass  | Pass  | Pass  | Pass  | Pass  | Pass  | Pass  | Pass  |
| Wetting time (sec)          | 18±2 | 15±1 | 12±2 | 10±3 | 5±1 | 4±4 | 3±1 | 3±6 |
| Water absorption ratio %    | 59.11±0.65 | 69.95±0.84 | 76.45±0.78 | 85.76±0.96 | 52.12±0.66 | 59.10±0.28 | 63.29±1.45 | 70.53±1.10 |
| In vitro disintegration time (sec) | 20±1 | 17±2 | 15±2 | 12±1 | 65±5 | 55±4 | 40±6 | 25±5 |
| In vivo disintegration time (sec) | 22±2 | 19±3 | 14±1 | 11±2 | 63±5 | 52±6 | 42±3 | 27±2 |
| Moisture uptake (%)         | 0.82±0.03 | 0.75±0.05 | 0.62±0.08 | 0.54±0.06 | 0.77±0.02 | 0.62±0.04 | 0.55±0.01 | 0.47±0.01 |
| Drug content (%)            | 98.24±0.15 | 98.45±0.20 | 97.98±0.55 | 97.12±0.66 | 95.75±1.03 | 96.59±0.56 | 98.22±0.87 | 97.27±0.92 |
| f₂                          | 72.54 | 77.74 | 88.16 | 97.21 | 71.13 | 87.55 | 81.49 | 71.80 |
In vitro nimesulide release was 95.13% (A1), 99.80% (A4), 91.58% (B1), and 94.21% (B4) batches of orodispersible tablets [Figures 6 and 7]. The similarity factor ($f_2$) is a logarithmic transformation of the sum-squared error of differences between the test $T_j$ and reference $R_j$ products over all time points. It is a useful tool for comparison of dissolution profiles when more than three or four dissolution time points are available.

$$f_2 = 50 \times \log \left\{ \left[ 1 + \frac{1}{n} \sum_{j=1}^{n} w_j \left( R_j - T_j \right) \right]^{2} - 0.5 \right\} \times 100$$

Where, $w_j$ is an optional weight factor. The similarity factor fits result between 0 and 100. It is 100 when the test and reference profiles are identical and tends to 0 as the dissimilarity increases. In order to consider similar dissolution profiles, $f_2$ values should be close to 100. The results obtained from the calculation of $f_2$ factor showed that there is a similarity of dissolution profiles between A1 to A4 and B1 to B4 and Nimulid MD tablets. However, A3 and A4 batches showed $f_2$ values of 88.16 and 97.21, respectively, which were highest amongst all the formulated batches.

Stability studies for the prepared batches containing Lallemantia reylenne as superdisintegrant was performed which indicated that there was no significant change in tablet hardness, friability, tensile strength, in vitro disintegration time, and drug content [Table 4].

CONCLUSION

In the present study the superdisintegrant property of Lallemantia reylenne was supported by the results of the in vitro disintegration study. The disintegration time of the prepared orodispersible tablets was found to be less than 60 seconds, which is desirable for disintegrating tablets. The moisture uptake study indicated no significant uptake of moisture by the prepared batches during the 10 day trial period. The percent moisture uptake was found to be 0.82 ± 0.03 to 0.54 ± 0.06 (A1-A4) and 0.77 ± 0.02 to 0.47 ± 0.07 (B1-B4).

The moisture uptake study indicated no significant uptake of moisture by the prepared batches during the 10 day trial period. Percent moisture uptake was found to be 0.82 ± 0.03 to 0.54 ± 0.06 (A1-A4) and 0.77 ± 0.02 to 0.47 ± 0.07 (B1-B4).
**Table 4: Stability study data of orodispersible tablets**

| Batch | Hardness | Friability | Tensile strength | Drug content | In vitro disintegration time |
|-------|----------|------------|------------------|--------------|----------------------------|
|       | 0 1 3   | 0 1 3     | 0 1 3            | 0 1 3        | 0 1 3                      |
| A1    | 2.84±   | 2.79±     | 2.52±            | 0.59±        | 0.61±                      | 0.63±                      | 0.457±                      | 0.449±                      | 0.432±                      | 98.24±                      | 98.10±                      | 97.74±                      | 20±1                      | 22±2                      | 24±1                      |
|       | 0.66    | 0.20      | 0.40             | 0.04         | 0.01                       | 0.03                       | 0.08                       | 0.05                       | 0.02                       | 0.15                       | 0.20                       | 0.15                       |                          |                          |                          |
| A2    | 3.40±   | 3.32±     | 3.27±            | 0.48±        | 0.51±                      | 0.55±                      | 0.541±                     | 0.535±                     | 0.530±                     | 98.45±                     | 98.42±                     | 98.36±                     | 17±2                      | 18±1                      | 20±3                      |
|       | 0.27    | 0.10      | 0.20             | 0.05         | 0.04                       | 0.07                       | 0.12                       | 0.10                       | 0.20                       | 0.20                       | 0.15                       | 0.55                       |                          |                          |                          |
| A3    | 4.10±   | 4.05±     | 3.95±            | 0.38±        | 0.41±                      | 0.43±                      | 0.654±                     | 0.648±                     | 0.636±                     | 97.98±                     | 97.72±                     | 96.65±                     | 15±2                      | 17±3                      | 19±1                      |
|       | 0.82    | 0.05      | 0.02             | 0.01         | 0.05                       | 0.09                       | 0.05                       | 0.02                       | 0.05                       | 0.55                       | 0.20                       | 0.35                       |                          |                          |                          |
| A4    | 4.80±   | 4.74±     | 4.69±            | 0.24±        | 0.27±                      | 0.29±                      | 0.760±                     | 0.751±                     | 0.743±                     | 97.12±                     | 96.85±                     | 96.52±                     | 12±1                      | 13±2                      | 15±1                      |
|       | 0.35    | 0.21      | 0.20             | 0.03         | 0.01                       | 0.02                       | 0.10                       | 0.03                       | 0.02                       | 0.66                       | 0.33                       | 0.14                       |                          |                          |                          |

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