Challenging to formulation through autonomous experiments and consideration of digital twin framework application

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1. Introduction

The application of autonomous experiments to digital twin frameworks has been studied through data-driven approaches based on statistical models and human skills. Data-driven approaches [1-4] have the advantage of inhering human experience as data by accumulating experimental data and knowledge. However, this approach can also be considered dependent on the implicit knowledge of experts, resulting in issues such as low reproducibility and unclear scalability.

In this paper, the grand design for applying this autonomous experiment framework to a digital twin framework are considered. The formulation that time-series condition transitions and experiments are paired with the formulation are proposed. While incorporating data-driven method, while time-series data on the emulsification process are acquired, annotated by correlating with phenomenology, and convert it into the information for autonomous experiment.

In addition, this paper shows the feasibility of autonomous digital twin framework aimed at "mixing" in industrial chemistry and materials for new material innovation.

2. Related work

Digital Twin Framework

The digital twin [5] framework is defined in the ISO 23247 series as a framework for the manufacturing industry. It is a purposeful digital representation of observable manufacturing elements, with the elements and digital representations appropriately synchronized. Framework for achieving specific goals such as monitoring, control, and optimization in manufacturing sites are defined.

As the application of this framework, the "formulation" process in new materials innovation and development are focused on. The "tacit knowledge" of expert formulation is relied on. Therefore, experiment data from "human" behavior, resulting in automated experiments that are focused on superficial human behavior are ued by general "Data-driven" method. However, there is a challenge in scalability for applications in processes that encompass non-superficial scope, such as materials research and development.

To evolve the digital twin framework for control and optimization, the feasibility study on an emulsification process is conducted. By acquiring "time-series data," the physics and chemistry of the process and considered its application to the framework are annotated.

Autonomous experiments devices

The experiment device for autonomous experiments in "mixing" and attempted to acquire time-series data are conducted. In addition, to annotate the definition of the mixing phenomenon are conducted by the measurement, and as the feasibility study, the definition of "mixed" based on the experimental results from the time-series data with the data acquired from the experimental device and chemical phenomena are annotated.

3. Positioning as digital twin

This section highlights the positioning of the digital twin in the digital twin context. In terms of the digital twin concept and its definition based on ISO 23247[6], the examining concepts, focusing on the DX for manufacturing industries outlined by Japan's Ministry of Economy, are focused on. The author has previously considered the application of the digital twin framework at each level [7]. Figure 1 indicates the associating DX/digital twin levels with processes.

In chemical innovation, "autonomous experimentation" is considered to be at Level 4, positioned as the optimization of the entire engineering chain based on data feedback. The engi-

neering chain is the chain of technologies leading up to product delivery, from planning to R&D, manufacturing, and after-sales service.

For realizing "Autonomous Experiments", while also utilizing a "data-driven" approach, it is believed that annotation with information from multiple domains is required. That's why Information from multiple domains includes not only human behavior in R&D, but also information on scientific theory and machine characteristics.

The providing feedback from "autonomous experimentation" to "autonomous behavior and the engineering chain" is a required function of a digital twin framework.

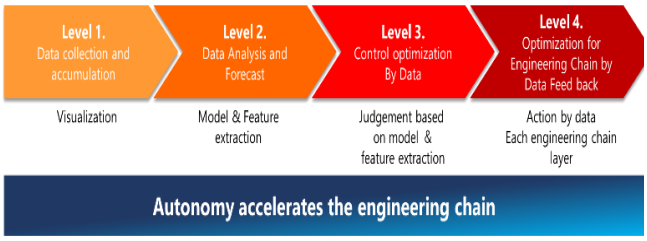

Fig. 1: Accelerating the Engineering Chain and Autonomous Experimentation

4. Experiment

As the experiment, the kneading device are developed and the experiment using emulsification as an example are conducted. The measurement data on the definition of "mixed" and its time series are obtained.

4.1 Experiments device

The experimental setup is shown in Figure 2. The test equipment is mainly composed of a mixier as experiment and a)Probe b)optical measurement device and and c)Rheometer as viscosity measurement.

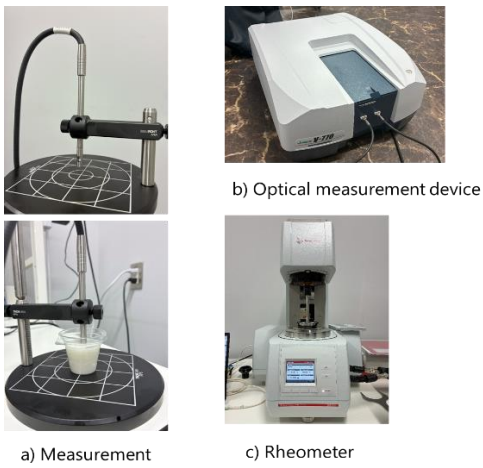

Fig. 2: Experimental set up

4.2 Formulation of mixing model

The experimental conditions are formulated as follows, and the measurement items are selected. The model formula is shown in Equation 1. We focus on energy flow.

$$dT/dt = P + Q_wall + Q_mix \quad (1)$$

The meaning of dT/dt is “Sensible heat term”, P is “Mechanical input”, Q_wall is “Heat exchange with wall Surface” and Q_mix is “Heat due to wetting, adsorption, cohesion, decohesion and interface formation”

4.3 Experimental Process

The experiments to mix and emulsify canola oil, polysorbate, and water are conducted. The controlling the rotation speed, and for each condition. The change in torque value and viscosity using a spectroscopic probe are observed. The temperature rise(Δ℃) is also measured after mixer.

5. Conclusion

In this research, to realize an autonomous digital twin framework aimed at "mixing" in the fields of industrial chemistry and materials for new material innovation are aimed.

1) The digital twin framework and positioning in accordance with ISO 23247 are demonstrated.

2) As an initial verification of autonomous experiments for the "mixing" process time-series data related to emulsification, and along with the data acquisition are aquired.

3) The physical and chemical aspects are calculated and annotated based on the formula.

As future challenges, the formulating the data including time-series state transitions, enabling dynamic decision-making for each condition are included.

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Appendix A. [Experimental Result]

Figure A.1 shows the experimental results. From left to right, the mixing times are 5, 10, 15, 20, 25, and 30 minutes. The condition transitions are confirmed by capturing the chemical phenomena that change over time. No separation occurred after 30 minutes of mixing are indicated. To be mixing disperses the particles overall and reduces the size of the oil molecules are considered. In terms of Research phase for new material innovation, in the process of combining with other materials, it will be necessary to combine them at intermediate condition, such as just completely mixed. Therefore, the reproducibility of intermediate stages is also required. Note that the experimental results are obtained one day later.

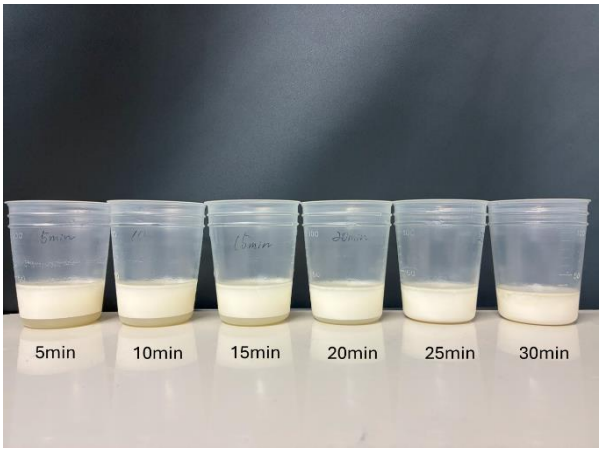

Fig. A.1: Experimental Result