Education for Chemical Engineers 36 (2021) 171–175

Contents lists available at ScienceDirect

Education for Chemical Engineers

journal homepage: www.elsevier.com/locate/ece

A remote foaming experiment

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ARTICLE INFO

Keywords:
Remote experiment
Polymer processing
Foaming

ABSTRACT

The 2019 pandemic locked lab doors to students and guest colleagues, with an impoverishment for all. Inspired by the 2015 work on "cloud chemistry" at the University of Nottingham, we designed an experimental facility to go beyond doors and boundaries (Skilton et al., 2015). On December 16th, 2020, an entire class of grad students virtually entered the foamlab of the University of Naples Federico II and remotely performed a plastic foaming experiment. The autoclave design, the plastic granules-handling robot, the process control and the vision acquisition system were all engineered to allow easy control by inexperienced operators and to guarantee safety to the local instructor. The digitalization of the foaming process is an exhaustive and modern pedagogical activity whereby chemical engineering and materials science and engineering students test their knowledge of transport phenomena, thermodynamics and process control. Students' response was enthusiastic: each of them felt at the centre of the active learning process.

1. Introduction

It is well known that a powerful way to improve long term learning in any teaching activity is based on direct active experimentation, and there are several works confirming the validity of this pedagogical approach (Selmer et al., 2007; Anastasio, 2015). However, laboratory access to students represents a critical issue, especially regarding safety, and the last couple of decades have seen a wealth of initiatives of remote- and cloud-based teaching.

Cloud chemistry was introduced by the group of Prof. M. Poliakoff in 2015 at the University of Nottingham, enlightening the scientific community with the vision of remote experimentation and the networking of chemists across the internet and the social, legal, safety, intellectual property, cybersecurity implications (Skilton et al., 2015). At that time, cloud experimentation meant sharing of experimental procedures among local and remote operators, followed by activities performed by the local operator, possibly supervised by the remote ones. Since then, with the introduction of robots, automatic experimentation and artificial intelligence flourished in materials science, with examples in chemistry, photovoltaic and life science (Steiner et al., 2019; MacLeod et al., 2019; Liu et al., 2013). More recently, robotic chemistry was introduced at the University of Liverpool by the group of Prof. A.I. Cooper, allowing mobile and intelligent robots striding the laboratory, to fast produce optimized formulations in a high-throughput fashion, without the need of an operator (Burger et al., 2020).

From a different perspective, the use of robots in the laboratory reduces the space left to human operation and the chances to learn. But the need for students to enter the laboratory, perform experiments, select the test conditions and strategy, even making mistakes, is stringent (Anon, 2021; Scanlon et al., 2004; Faulconer and Gruss, 2018). As instance, operating complex and possibly dangerous processing equipment typical of industrial manufacturing plants is an essential portion of their education. We report a first of a kind experiment performed by students from their homes in the university laboratory, to produce plastic foams by operating a high-pressure and high-temperature gas foaming equipment. Students were voice-guided by the instructor to perform all of the processing steps required to produce the foam from the very polymer granule to the final plastic foams, with no physical intervention by the instructor.

2. Experiment description

Conceiving an experiment to be entirely carried out remotely requires reinventing the equipment and the process from scratch. Operations that would be too difficult to perform with a (cheap) robot have to be redesigned and/or replaced, possibly with adjustment of the whole

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https://doi.org/10.1016/j.ece.2021.05.003
Received 22 March 2021; Received in revised form 29 May 2021; Accepted 30 May 2021
Available online 6 June 2021
1749-7728/

procedure. Details of the adopted hardware and software, along with the test procedure are provided in the Supplementary Material. Here, it is worth citing the closure of the autoclave as an example of the equipment design process. Foaming autoclaves are typically pressure-tight secured by bolts and screws, which have to be fastened in the correct sequence and torque. Availability of a suitable robot able to do so is extremely difficult and expensive, also requiring extensive training. We instead utilized a hydraulic press to apply the clamping force to the autoclave. Autoclave closing and opening is then attained via pc-operated electro-valves. Fig. 1 reports pictures of the equipment, showing the autoclave with temperature and pressure sensors, the valves for gas dosing, the hydraulic press, the handling robot and the cameras (the costs of the pieces of equipment are reported in Fig. S1).

The 'Remote Gas Foaming' lectures took place in the foamlab at the University of Naples Federico II. Microsoft Teams from Microsoft (Redmond, USA) was used to allow remote control of the equipment and perform the experiment. LabVIEW® from National Instruments (N Mopac Expwy Austin, TX, USA) was used to control the robotic arm; GF Express® from Gefran S.p.A. (Provaglio d'Iseo, Brescia, Italy) enabled the valves operation, the process temperature and pressure control (Fig. 2). Data flow, consent for activating buttons and controls and safety procedures were implemented to conduct the experiment by non-expert user. Fig. S2 shows a scheme of the sequential logic operations; Fig. 3 shows selected images of the sequence of operations.

The experiment took about 2 h. During the first half hour, students maneuvered the robot to place the polymer granule into the wells of the autoclave (Phase I). The use of the 3D camera was useful, but not necessary, as students proved skilled, comfortable and accustomed to orientation by 2D view. The 150$ robot and suction head proved perfectly fine for granule manipulation. After having checked the correct granule positioning and, in particular, that the autoclave was clear from granules accidentally misplaced (thus hindering the correct closure and damaging the equipment), Phase II of autoclave closure was conducted, by activating the "close autoclave button". Foam enthusiasts would love the simplicity of the procedure, in particular skipping of lengthy and tiresome bolt closure procedure. Phase III consisted in the very foaming process, conducted in this case on polycaprolactone (PCL), a biodegradable polyester with carbon dioxide as the blowing agent. Specifically, the temperature and pressure programs were aimed at: melting the polymer (at, 80 °C, considering the melting temperature of PCL =60 °C), solubilize the blowing agent (CO_2 pressure of 40 bar), bring the system to the foaming temperature (typically, few degrees above solidification temperature, 40 °C in our case) and finally pressure quench at 10 MPa/s. Phases II and III were actuated and monitored from the foaming process control panel (bottom in Fig. 2) and took about 1 h (most of which required for the CO_2 solubilization in PCL at constant pressure and temperature). After pressure quench, the autoclave was opened and foamed samples collected for further testing (Phase IV - 1/2 h).

3. Learning & education

The remote foaming experiment was designed as a laboratory course facility. Laboratory classes have been taught in the period December 2020 - May 2021 to students from different Universities and programs: i) Soft Matter Engineering course, M.Sc. in Chemical Engineering at the University of Naples Federico II, Italy; ii) Polymer Processing course, M. Sc. in Materials Engineering at the University of Naples Federico II, Italy; iii) Nanobiomaterials course, M.Sc. in Biotechnology at the Tecnológico de Monterrey, Mexico; iv) Selected Topics in Bioprocesses course, M.Sc. in Biotechnology at the Tecnológico de Monterrey, Mexico. Students were exposed to the remote experimental session after a specific lecture on foams and foaming applications, where the relevant background on thermodynamics, transport phenomena and process control were introduced to analyse and model the key steps in foaming. In the preparatory lecture, foam properties and morphologies were presented, together with the foaming processes and process variables. The lecture and experimental session of the foaming process took place on the

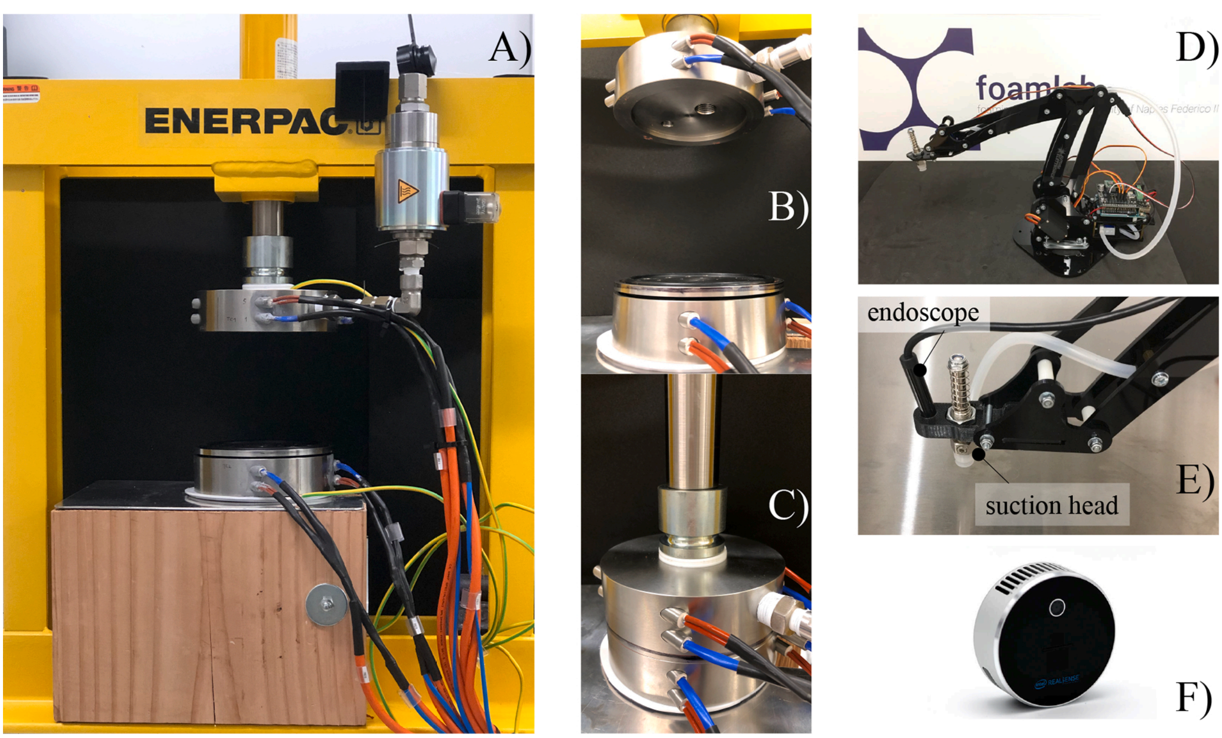

Fig. 1. Remote foaming equipment. A) open autoclave on the wooden base, with heating cartridges and connection to the gas dosing valve; the yellow frame is the hydraulic press for pressure-tight closing the autoclave. B) body and lid of the open autoclave; the lower surface of the lid shows the ports to gas dosing and gas evacuation. C) body and lid of the closed autoclave. D) robotic arm. E) robot hand with suction head and endoscope camera. F) global camera for 3D view with LiDAR technology.

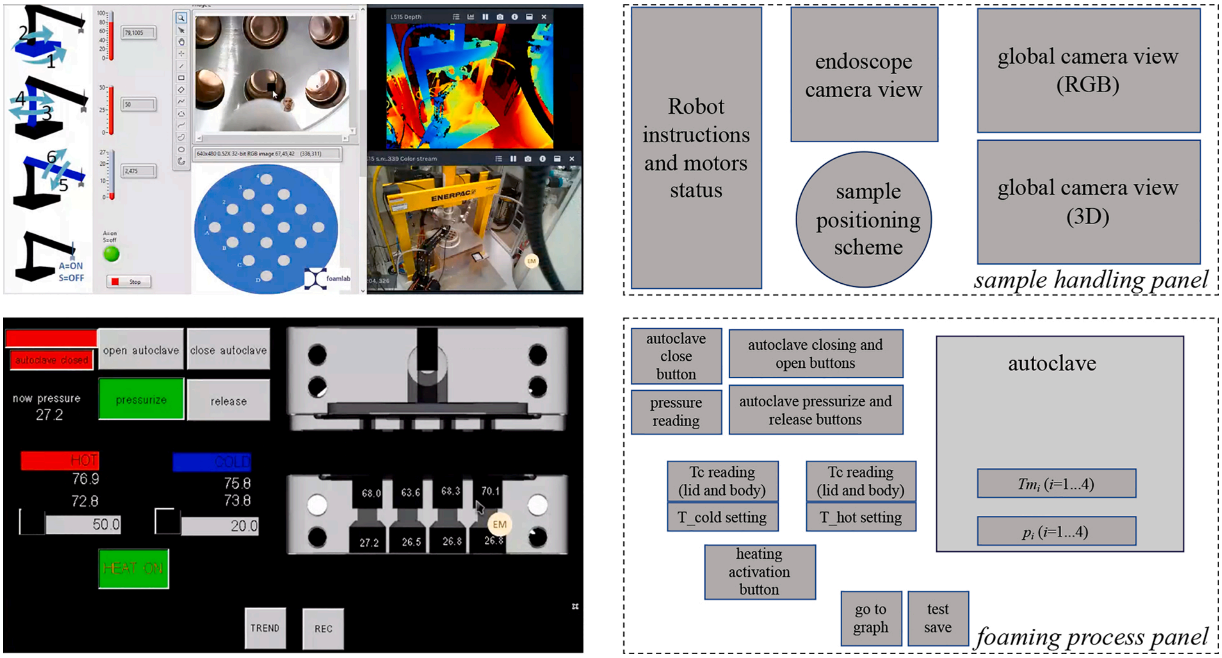

Fig. 2. Left: graphic user interface during operations when handling the sample (LabVIEW® window - up) and when operating the autoclave (Gefran window - down). The global camera is active during the whole experiment showing a depth colour image (3D view) and a 2D view of the apparatus. Right: description of the corresponding parts of the GUIs.

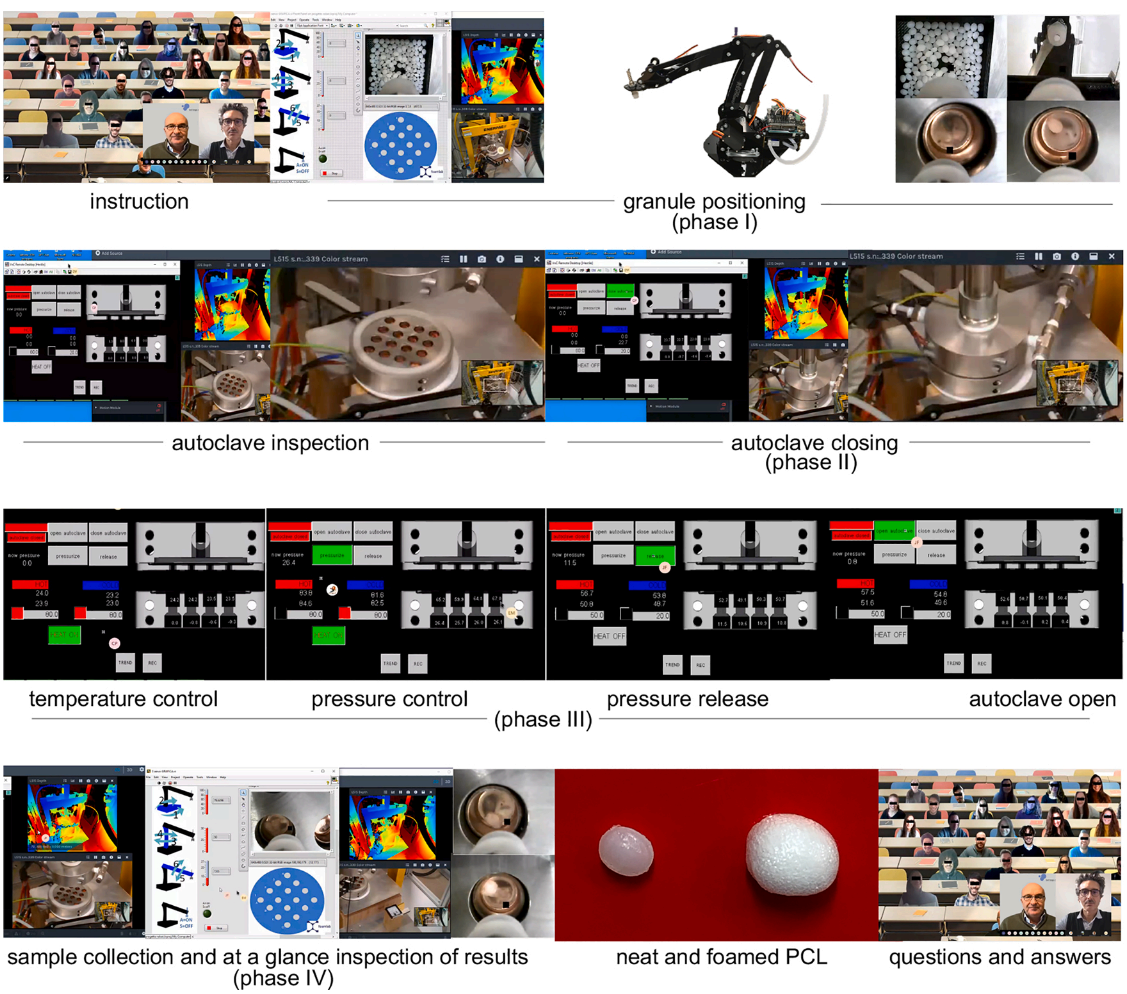

Fig. 3. Sequence of operations performed during the remote laboratory lecture.

Microsoft Teams workspace. The software allows both control from a remote computer and screen sharing to all of the meeting participants. The students were asked to discuss critically with each other during the virtual meeting on how to select the experimental conditions. Namely, process parameters are sorption temperature and pressure, sorption time to attain an equilibrium polymer/gas solution and foaming temperature. Indeed, the mutual diffusivity of the blowing agent within the polymer is dependent on their nature, the temperature and the blowing agent concentration. The instructor oversaw the discussion and guided them toward the optimal solution, suggesting the pieces of literature providing the required data. Students had to choose the temperature compromise to accelerate the sorption process while ensuring safety operation and avoiding thermal degradation of the polymer. They also learned how solubility could depend on the temperature and pressure, and which are the mechanisms involved in the bubble nucleation and growth to proper select the foaming temperature. They set the PID control parameters to prevent the heating cartridges from overheating the autoclave and the samples, with hints on process control and the notions of "control" and "melt" temperatures in a processing equipment. To select the proper foaming temperature, they discussed about the available setting mechanisms (PCL is a semi-crystalline polymer) and the plasticization effect by the gas also in view of the thermal history experienced by the polymer during the test. Finally, they discussed the outcomes of the experiment after having collected the foamed samples from the wells. The granules positioning was performed by 16 students, being the autoclave constituted of 16 wells. 16 different students collected the foamed samples after foaming. The autoclave control stages were also conducted by different students, which took turns in the closing of the autoclave, pressurization, temperature program setting, pressure release and autoclave opening. This approach proved effective in encouraging active remote participation. The students voluntarily participated to the experiment without a pre-defined order. Local instructors were physically present in the lab to guide the students, but without physical intervention, unless required for safety or malfunctioning.

Video tutorials are becoming a standard in the learning process of the new digital era, but they still lack active learning features. This is the ultimate goal of the foaming experiment in the context of Chemical Engineering and Materials Science and Engineering. By conducting it remotely, distance barriers were eliminated and the students, not the experiment itself, became the main focus of the lecture. They proved already skilled to control the robot without making gross mistakes, so the local instructor did not provide them with further help such as indicating the position of each well with respect to the robotic arm degrees of freedom (see Table S3 –*Supplementary Material*). The robotic arm along with the vision acquisition setup transformed this experience in a *serious game* whereby even the shyest students engaged enthusiastically with the activity (Alvarez and Djaouti, 2014). We realized that the association of teaching and entertainment could help the students focus on the lecture. The whole remote foaming test was completed with success. Some students expressed excitement and surprise for the unusual learning experience. Virtual reality would certainly improve even more this active learning activity. At the end of the pick and place operation, the students were asked to propose improvements of the apparatus. Not surprisingly, they suggested to automatize the pick and place operation by forcing the robotic arm to move to the specific wells' position. This upgrade would certainly transform the experiment in a format similar to a videotutorial and, in view of the considerations explained above, it will not be implemented for the time being.

After the completion of the operations, students were asked to complete an anonymous satisfaction survey about the remote experimentation (see *Supplementary Material* for more details). This activity also promoted a better understanding of the covered topics and the theoretical background deficiencies. In summary, the robot and the process control (both hardware and software) as well as the thermodynamics of polymers/gas solutions (specifically the polymer thermal properties and its behaviour in contact with a gas) were seen as the most difficult topics treated during the experiment. Almost 90 % of the interviewees reported their theoretical background was sufficient to follow and understand the operations. 100 % of them found the discussion about the definition of the operating conditions useful. The active learning experience was indeed a success: 80 % of the participants felt they actively took part to the experimentation and effectively participated by conducting, for example, the pick and place operation or the autoclave closure/opening and process control steps. 67 % of the participants also confirmed to have taken part to the definition of the experimental conditions. Distance learning and remote experimentation were seen as good practices by the majority, especially because of the pandemic. Nevertheless, most of them would rather conduct the experiment on-site. In the participants' opinion, remote experimentation is a good practice to complement the theoretical course allowing them to deal with real case studies. They suggested to add more cameras to improve the 3D view of the apparatus. Some of them found the experience too slow, and thus sometime boring. We agree that waiting for the sorption time remotely, as well as for the system to equilibrate to the prescribed temperature is boring, but this is unavoidable in the (real) lab. Some students experienced a lag in the robot control.

4. Process safety

The alignment of academia and industrial engineering has led to an increasing interest in process safety to integrate students' curriculum (Luo and Westmoreland, 2015c). Several safety considerations were addressed to construct the apparatus and were transferred to the students during the lecture. First of all, safety standards such as UNI or ASME do not usually apply to prototypes and the inventor must evaluate the risks related to the system usage. To this aim, we calculated first the maximum gas pressure at which the autoclave could operate (approximately 50 bar), considering the press tonnage and the size of the autoclave. Possible failure of the sealing gaskets placed between the two autoclave parts would inevitably harm the local operator. Consequently, a polycarbonate barrier surrounding the whole apparatus was installed. It is also mandatory wearing ear defenders for everyone working nearby the autoclave during operation, especially during the stages involving high pressure and during the pressure release. CO_2 leakage is not toxic but could lower the oxygen concentration in the lab, so an atmospheric oxygen concentration sensor with a built-in audio alarm was installed as well. Remote operation inevitably reduces operation risks: the remote operator does not need to wear any personal protective equipment and the robotic arm prevents him from using his own hands. For instance, the risk of crushing deriving from the failure of the screw connecting the autoclave upper part with the pump moving piston was completely eliminated. At the present stage, the temperature must not reach values higher than 120 °C to prevent the wooden base from being damaged. This part of the apparatus will be replaced with an aluminium base to increase the temperature maximum limit. GF Express® software package allows the installation of audio alarms or automatic execution stops. At the present time no safety temperature, pressures and time limits were added. Maximum pressure was imposed by the line pressure. Conversely, we decided to add a specific feature to the process control program preventing the opening of the autoclave when the pressure inside it is higher than 1.5 bar.

University lab rules apply and the walkable area in our lab only allows a maximum of 5 people to assist in person the foaming experiment. By conducting the experiment remotely, already 100 students (maximum 30 per class) took active part to the remote foaming experiment. The students particularly appreciated having the same perspective from their homes. The apparatus can be easily modified to add new features, for instance allowing a morphological characterization of the foamed samples.

5. Conclusions

A remote foaming experiment was successfully conducted by students of different courses of the University of Naples Federico II, Italy and of the Tecnológico de Monterrey, Mexico. The events were part of the teaching activities digitalization by now mandatory due to the pandemic. The students actively participated to the experiment, testing their knowledge of thermodynamics, transport phenomena and process control. They cooperated in choosing the optimum values of the process parameters. We believe this experience has to be shared among teachers, to foster the development of other remote experiments and expedite the process of providing students with active learning tools which would complete their education in scientific fields. The foamlab at University of Naples Federico II is already looking for classes, anywhere in the world, to share this first case. Remote foaming is not only a teaching opportunity. Scientists all over the world could benefit from this technique opening the path for future collaborations and improvements of the experimental apparatus. We believe this new way of doing research would reduce inequalities between scientists all over the world, as in the Nottingham vision.

Author contributions

EDM and PLM conceived the project, EDM, DT designed the equipment; VL designed, installed and programmed the robot, AL designed the workflow and assisted students teaching. EDM, VL, AL and DT wrote the paper.

Data and materials availability

Video of the whole remote foaming experiment can be shared upon request.

Declaration of Competing Interest

The authors report no declarations of interest.

Acknowledgments

Admin staff of the University of Naples Federico II, which were supportive, during COVID19 lockdown, in buying the pieces of equipment. G. Buonocore (Sonepar S.p.A.) for assistance with Gefran equipment.

Appendix A. Supplementary data

Supplementary Material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.ece.2021.05.003.

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